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PLATTNER'S MANUAL

OF

QUALITATIVE AND QUANTITATIVE ANALYSIS WITH THE BLOWPIPE.

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OF

QUALITATIVE AND QUANTITATIVE

ANALYSIS WITH THE BLOWPIPE.

FROM THE LAST GERMAN EDITION.

REVISED AND ENLARGED

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EDITED BY

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WITH NUMEROUS ILLUSTRATIONS.

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PREFACE.

A WORK like the present needs but a short preface. A good book on the BLOWPIPE has long been wanted in England, and it is because of the increasing importance of analysis by means of this instrument, that I have edited the great work of Plattner.

While staying, last year, at the Freiberg Mining Academy, Saxony, I was so impressed with the perfection to which the use of the Blowpipe has been brought by the German teachers, who use Plattner's work as a text-book, that I resolved to give English students the benefit of Plattner's researches.

The first English translation of this work was issued in New York, and I have followed generally the translation of the American edition, omitting, however, some few portions which I have thought superfluous. I have also omitted the long list of minerals given under each heading, as iron, lead, &c., amounting to several hundreds in the German edition. However useful such lists may be as a mineralogical reference, still they hardly belong to the province of the blowpipe. The headings of the different combinations, and all the principal minerals, however, have been retained and their characteristics fully described as in the German original. I have added a new drawing and description of a mechanical blowpipe, which is the only one which is at once portable and thoroughly simple and effective. It would not have been a very laborious or difficult task for me to have greatly reduced the size of the book, but I have thought it better, as Plattner may be justly called the father of this department of analysis, to edit the work in its entirety.

In commending the volume to the English student, I need only add that it is by far the most complete work extant on a subject both of growing practical importance and of extreme interest.

T. HUGO COOKESLEY.

LONDON, *April*, 1875.

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In consequence of the exceedingly brittle nature of the type-metal which has been used to print the formulas in this book, *e.g.* $\rlap{-}\mathrm{F}$, W, $\overset{'''}{\mathrm{S}}\mathrm{b}$, &c., it has been found impossible to prevent some few of the points breaking off during the process of printing. As it is improbable that all these points broke off *simultaneously*, one errata sheet would obviously not suit all the copies of the edition. I have thought it better then to append a list of all the formulas in the book, so that the reader can at once refer to their respective pages, and fill in with pen and ink any deficiencies which may occur. It will only take him an hour's trouble at the most.

Page	101	section	4	under	Strontia	read	$\dot{\mathrm{Sr}}$, $\dot{\mathrm{S}}\mathrm{r}\ \mathrm{C}$
,,	,,	,,	1	,,	Lime	,,	Ca, Ca C
,,	,,	,,	2	,,	,,	,,	Ca C
,,	103	,,	1	,,	Antimony	,,	Sb
,,	,,	,,	3	,,	,,	,,	Sb
,,	,,	,,	5	,,	,,	,,	$\dot{\mathrm{Sb}}$

LIST OF FORMULÆ.

Page	105	section	1	under	Cerium	read	$\dddot{\text{Ȼe}}$
,,	,,	,,	2	,,	Chromium	,,	$\dddot{\text{Ȼr}}$
,,	,,	,,	4	,,	,,	,,	$\dddot{\text{Ȼr}}$, $\dddot{\text{Ȼr}}$
,,	107	,,	2	,,	Iron	,,	$\dot{\mathrm{Fe}}$, $\dddot{\text{Ꞙe}}$
,,	109	,,	3	,,	Molybdenum	,,	$\ddot{\mathrm{Mo}}$
,,	110	,,	1	,,	,,	,,	$\ddot{\mathrm{Mo}}$
,,	,,	,,	2	,,	,,	,,	$\ddot{\mathrm{Mo}}$
,,	,,	,,	,,	,,	,,	,,	$\ddot{\mathrm{Mo}}$
,,	111	,,	1	,,	Osmium	,,	$\ddddot{\mathrm{Os}}$
,,	113	,,	1	,,	Tellurium	,,	$\ddot{\mathrm{Te}}$
,,	,,	,,	2	,,	,,	,,	$\ddot{\mathrm{Te}}$
,,	,,	,,	4	,,	,,	,,	$\ddot{\mathrm{Te}}$
,,	,,	,,	1	,,	Tin	,,	$\ddot{\mathrm{Sn}}$, Sn
,,	114	,,	1	,,	Tungsten	,,	$\ddot{\mathrm{W}}$
,,	,,	,,	2	,,	,,	,,	$\ddot{\mathrm{W}}$
,,	,,	,,	3	,,	,,	,,	$(\ddot{\mathrm{W}}.\ddot{\mathrm{W}})\,\ddot{\mathrm{W}}$, $\ddot{\mathrm{Ti}}$
,,	115	,,	1	,,	Uranium	,,	$(\dot{\mathrm{U}}\,\dddot{\mathrm{U}})$
,,	,,	,,	2	,,	,,	,,	$\dddot{\text{Ꞙe}}$, $(\dot{\mathrm{U}}\,\dot{\mathrm{U}})$, $\dot{\mathrm{U}}$
,,	,,	,,	3	,,	,,	,,	$(\dot{\mathrm{U}}\,\dddot{\mathrm{U}})$, $\dot{\mathrm{U}}$
,,	,,	,,	1	,,	Vanadium	,,	$\dot{\mathrm{V}}$
,,	,,	,,	2	,,	,,	,,	$\ddot{\mathrm{V}}$

Page	134	line	14	read	$\dot{\mathrm{Ca}}\,\dddot{\mathrm{S}}$
,,	136	,,	28	,,	$\dot{\mathrm{Ba}}\,\dot{\mathrm{S}}$, $\dot{\mathrm{Sr}}\,\dddot{\mathrm{S}}$
,,	137	,,	27	,,	$\dot{\mathrm{Ca}}\,\dddot{\text{Ꞙe}}$
,,	138	,,	13	,,	$\dot{\mathrm{Ca}}\,\ddot{\text{Ꞙe}}$
,,	140	,,	30	,,	$\dot{\mathrm{Ca}}\,\dddot{\mathrm{S}}$, $\dot{\mathrm{Mg}}\,\dddot{\mathrm{S}}$, $\dot{\mathrm{K}}\,\dot{\mathrm{S}}$

Page 141 line 11 read $\dot{\mathrm{Ca}}\,\ddot{\mathrm{S}}$, $\dot{\mathrm{Na}}\,\dddot{\mathrm{S}}$

,, 150 ,, 12 ,, $\dot{\mathrm{Y}}$, $\ddot{\bar{\mathrm{A}}}\mathrm{l}$, $\dddot{\bar{\mathrm{F}}}\mathrm{e}$, $\dot{\mathrm{Ca}}$, $\dot{\mathrm{Mg}}$, $\dot{\mathrm{Mn}}$

,, ,, ,, 14 ,, $\dot{\mathrm{Y}}$, $\bar{\mathrm{F}}\mathrm{e}$, $\dddot{\bar{\mathrm{A}}}\mathrm{l}$.

,, 154 ,, 7 ,, $\dot{\mathrm{Mg}}$, $\dot{\mathrm{Fe}}$, $\dot{\mathrm{Mn}}$.

,, 158 ,, 26 ,, $\ddot{\mathrm{Si}}$, $\bar{\mathrm{F}}\mathrm{e}$, $\dot{\mathrm{Ca}}$

,, 175 ,, 1 ,, $\dot{\mathrm{Ca}}$, $\dot{\mathrm{Y}}$, $\dot{\mathrm{Ce}}$, $\dot{\mathrm{La}}$, $\dddot{\bar{\mathrm{F}}}\mathrm{e}$, $\dot{\mathrm{Mn}}$, $\bar{\mathrm{U}}$

,, ,, ,, 3 ,, $\dddot{\mathrm{Ta}}$, $\ddot{\mathrm{Nb}}$, W, $\ddot{\mathrm{Sn}}$

,, ,, ,, 4 ,, $\ddot{\mathrm{Ti}}$, $\dot{\mathrm{Zr}}$, $\dot{\mathrm{Th}}$

,, ,, ,, 10 ,, $\ddot{\mathrm{Nb}}$, $\dot{\mathrm{Ta}}$, $\ddot{\mathrm{W}}$, $\ddot{\mathrm{Ti}}$, $\ddot{\mathrm{Zr}}$.

,, ,, ,, 11 ,, $\ddot{\mathrm{Th}}$, $\ddot{\mathrm{Sn}}$

,, ,, ,, 21 ,, $\dot{\mathrm{Ta}}$, $\ddot{\mathrm{Nb}}$, W, $\ddot{\mathrm{Th}}$, $\ddot{\mathrm{Sn}}$

,, ,, ,, 22 ,, $\ddot{\mathrm{Ti}}$, $\ddot{\mathrm{Zr}}$

,, ,, ,, 29 ,, $\dddot{\mathrm{Ta}}$, $\ddot{\mathrm{Nb}}$, $\ddot{\mathrm{Th}}$, $\ddot{\mathrm{W}}$.

,, ,, ,, 30 ,, $\ddot{\mathrm{Sn}}$

,, 176 ,, 20 ,, $\dddot{\mathrm{Ta}}$, $\ddot{\mathrm{Nb}}$

,, ,, ,, 21 ,, $\ddot{\mathrm{W}}$, $\ddot{\mathrm{Ti}}$, $\ddot{\mathrm{Zr}}$, $\ddot{\mathrm{Sn}}$

,, ,, ,, 34 ,, $\ddot{\mathrm{Ti}}$, $\ddot{\mathrm{Zr}}$, $\ddot{\mathrm{Th}}$, $\ddot{\mathrm{Ti}}$, Y, $\ddot{\mathrm{Th}}$

,, ,, ,, 35 ,, $\dot{\mathrm{Zr}}$, $\dot{\mathrm{Ce}}$, $\dot{\mathrm{La}}$, $\dddot{\bar{\mathrm{F}}}\mathrm{e}$, $\bar{\mathrm{U}}$, $\dot{\mathrm{Ca}}$, $\dot{\mathrm{Mn}}$

,, 177 ,, 19 ,, Y, $\dot{\bar{\mathrm{F}}}\mathrm{e}$, $\dddot{\bar{\mathrm{U}}}$

,, ,, ,, 30 ,, $\dot{\mathrm{Y}}$, $\dddot{\bar{\mathrm{F}}}$, $\dddot{\bar{\mathrm{U}}}$

,, ,, ,, 35 ,, $\bar{\mathrm{F}}\mathrm{e}$

,, ,, ,, 36 ,, $\ddot{\bar{\mathrm{U}}}$

,, ,, ,, 38 ,, $\dot{\bar{\mathrm{F}}}\mathrm{e}$, $\dddot{\bar{\mathrm{U}}}$

,, 178 ,, 3 ,, $\ddot{\bar{\mathrm{U}}}$

,, ,, ,, 4 ,, $\dddot{\bar{\mathrm{F}}}\mathrm{e}$, $\dddot{\bar{\mathrm{U}}}$

,, 180 ,, 16 ,, $\dot{\mathrm{Ca}}$, $\dot{\mathrm{Mg}}$, $\dddot{\bar{\mathrm{A}}}\mathrm{l}$, $\dot{\mathrm{Be}}$, $\dot{\mathrm{Y}}$, $\dot{\mathrm{Ce}}$, $\dot{\mathrm{La}}$, $\dot{\bar{\mathrm{F}}}\mathrm{e}$, $\dot{\mathrm{Mn}}$, $\dot{\mathrm{K}}$, $\dot{\mathrm{Na}}$

,, ,, ,, 18 ,, $\ddot{\bar{\mathrm{A}}}\mathrm{l}$, $\dot{\mathrm{Be}}$, $\dot{\mathrm{Y}}$, $\dot{\mathrm{Ce}}$, $\dot{\mathrm{La}}$, $\bar{\mathrm{F}}\mathrm{e}$

,, ,, ,, 19 ,, $\dot{\mathrm{Ca}}$, $\dot{\mathrm{Mg}}$, $\dot{\mathrm{Mn}}$.

,, ,, ,, 29 ,, $\dddot{\bar{\mathrm{A}}}\mathrm{l}$, $\dot{\mathrm{Be}}$.

LIST OF FORMULÆ.

Page 180 line 33 read $\dot{\mathrm{Y}}$, $\dot{\mathrm{Ce}}$, $\dot{\mathrm{La}}$, $\mathrm{\rlap{-}Fe}$.

,, ,, ,, 34 ,, $\dddot{\mathrm{\rlap{-}Fe}}$

,, 181 ,, 17 ,, $\dot{\mathrm{Na}}$, $\dot{\mathrm{Ca}}$, $\dot{\mathrm{Mg}}$, $\ddot{\mathrm{Zr}}$, $\ddot{\mathrm{\rlap{-}Fc}}$, $\dot{\mathrm{Mn}}$, $\ddot{\mathrm{Si}}$, $\overset{\cdot\cdot\cdot\cdot\cdot}{\mathrm{\rlap{-}Nb}}$

,, ,, ,, 32 ,, $\ddot{\mathrm{Zr}}$, $\dddot{\mathrm{\rlap{-}Fe}}$, $\dot{\mathrm{Ca}}$.

,, ,, ,, 34 ,, $\ddot{\mathrm{Zr}}$, $\dot{\mathrm{\rlap{-}Fe}}$.

,, 182 ,, 2 ,, $\ddot{\mathrm{Zr}}$, $\ddot{\mathrm{\rlap{-}Fe}}$

,, ,, ,, 6 ,, $\dddot{\mathrm{\rlap{-}F}}$.

,, ,, ,, 9 ,, $\ddot{\mathrm{Zr}}$, $\dddot{\mathrm{\rlap{-}Fe}}$.

,, ,, ,, 16 ,, $\dot{\mathrm{Na}}$, $\dot{\mathrm{Ca}}$, $\dot{\mathrm{Mg}}$, $\dot{\mathrm{Mn}}$, $\dot{\mathrm{Ca}}$

,, ,, ,, 17 ,, $\dot{\mathrm{Mg}}$, $\dot{\mathrm{Mn}}$

,, 183 ,, 8 ,, $\ddot{\mathrm{Zr}}$, $\dot{\mathrm{Ca}}$, $\dot{\mathrm{Fe}}$.

,, ,, ,, 25 ,, $\ddot{\mathrm{Zr}}$, $\dot{\mathrm{Ca}}$.

,, ,, ,, 26 ,, $\dot{\mathrm{Mg}}$, $\dddot{\mathrm{\rlap{-}Fc}}$.

,, ,, ,, 27 ,, $\ddot{\mathrm{Zr}}$, $\dddot{\mathrm{\rlap{-}Fe}}$, $\dot{\mathrm{Ca}}$

,, ,, ,, 28 ,, $\dot{\mathrm{Mg}}$.

,, 191 ,, 33 ,, $\ddot{\mathrm{\rlap{-}Fe}}$, $\dot{\mathrm{Ca}}$, $\dot{\mathrm{La}}$, $\dot{\mathrm{Di}}$, $\dddot{\mathrm{\rlap{-}Al}}$.

,, ,, ,, 34 ,, $\dot{\mathrm{Ca}}$, $\dot{\mathrm{Mg}}$, $\dot{\mathrm{Mn}}$.

,, ,, ,, 36 ,, $\dddot{\mathrm{\rlap{-}Al}}$

,, ,, ,, 38 ,, $\dddot{\mathrm{\rlap{-}Al}}$

,, 196 ,, 27 ,, $\dot{\mathrm{Co}}^3\,\overset{\cdot\cdot\cdot\cdot\cdot}{\mathrm{As}}$.

,, 199 ,, 34 ,, $\overset{\prime}{\mathrm{Fe}}$.

,, ,, ,, 37 ,, $\overset{\prime}{\mathrm{Ag}}$, $\overset{\prime}{\mathrm{\rlap{-}Cu}}$.

,, 200 ,, 4 ,, $\overset{\prime}{\mathrm{\rlap{-}Cu}}$, $\overset{\prime}{\mathrm{Fe}}$, $\overset{\prime}{\mathrm{\rlap{-}Cu}}$, $\overset{\prime}{\mathrm{\rlap{-}Fe}}$. $\overset{\prime}{\mathrm{Fe}}$.

,, ,, ,, 9 ,, $\overset{\prime}{\mathrm{Pb}}$.

,, ,, ,, 10 ,, $\overset{\prime}{\mathrm{\rlap{-}Fe}}$, $\overset{\prime}{\mathrm{Zn}}$.

,, ,, ,, 11 ,, $\mathrm{S}'''\mathrm{b}$, $\overset{\prime}{\mathrm{Ag}}$.

,, 214 ,, 32 ,, $\overset{\prime}{\mathrm{Pb}}$, $\mathrm{S}'''\mathrm{b}$

,, 215 ,, 22 ,, $\overset{\prime}{\mathrm{\rlap{-}Cu}}$

,, 240 ,, 38 ,, $\dddot{\mathrm{Sb}}$

LIST OF FORMULÆ.

Page 241 line 4 read $\acute{\text{P}}\text{b}$

,, 242 ,, 16 ,, $\text{Pb}, \overset{\cdot\cdot}{\dddot{\text{S}}}\text{b}, \dot{\text{P}}\text{b}, \dddot{\text{S}}, \dot{\text{P}}\text{b}.$

,, 244 ,, 18 ,, $\dot{\text{P}}\text{b}, \ddot{\text{S}}.$

,, 260 ,, 34 ,, $\ddot{\text{T}}\text{e}$

,, ,, ,, 37 ,, $\ddot{\text{S}}\text{i}$

,, 267 ,, 35 ,, $\text{S}\overset{\prime\prime\prime}{\text{b}}, \text{A}\overset{\prime\prime\prime}{\text{s}}$

,, ,, ,, 37 ,, $\text{H}\acute{\text{g}}$

,, 268 ,, 2 ,, $\text{H}\acute{\text{g}}$

,, ,, ,, 4 ,, $\text{H}\acute{\text{g}}$

,, 271 ,, 10 ,, $\dot{\text{Ȼ}}\text{u}$

,, 281 ,, 31 ,, $\text{A}\overset{\prime\prime\prime}{\text{s}}$

,, 282 ,, 9 ,, $\text{S}\overset{\prime\prime\prime}{\text{b}}, \text{A}\overset{\prime\prime\prime}{\text{s}}$

,, ,, ,, 12 ,, $\text{S}^{\prime\prime\prime}\text{b}$

,, ,, ,, 28 ,, $\text{A}\overset{\prime\prime\prime}{\text{s}}, \text{S}^{\prime\prime\prime}\text{b}$

,, 285 ,, 27 ,, $\dot{\text{I}}\text{r}, \dot{\text{O}}\text{s}, \dot{\text{F}}\text{e}, \dddot{\text{Ɨ}}\text{r}\ \text{Ɵ}^{\prime\prime\prime}\text{S}\ \dddot{\text{Ȼ}}\text{r}$

,, 298 ,, 13 ,, $\text{S}^{\prime\prime\prime}\text{b}$

,, ,, ,, 22 ,, $\text{S}^{\prime\prime\prime}\text{b}$

,, ,, ,, 24 ,, $\overset{\cdot\cdot}{\dddot{\text{S}}}\text{b}$

,, 325 ,, 1 ,, $\ddot{\text{T}}\text{e}$

,, 339 ,, 26 ,, $\text{F}\overset{\prime\prime}{\text{e}}, \overset{\prime\prime\prime}{\text{Ꞙ}}\text{e}, \overset{\prime\prime}{\text{M}}\text{n}, \acute{\text{C}}\text{u}.$

,, ,, ,, 32 ,, $\text{S}^{\prime\prime\prime}\text{b}, \text{S}^{\cdots}\text{b}$

,, 355 ,, 29 ,, $\dot{\text{K}}\ \dot{\text{C}}\text{y}$

,, ,, ,, 30 ,, $\dot{\text{K}}\ \ddot{\text{C}}$

,, 487 ,, 28 ,, $\dot{\text{K}}\ \dddot{\text{S}} + \dddot{\text{Ȼ}}\text{r}$

,, 493 ,, 8 ,, $\dot{\text{K}}\ \dddot{\text{S}} + \dddot{\text{Ȼ}}\text{r}$

,, 379 ,, 6 ,, $0.32 + \frac{0.36 - 0.32}{3}$

,, ,, ,, 8 ,, $0.33 = 53.78$ per cent.

SECTION I.

DESCRIPTION OF APPARATUS AND REAGENTS.

I. THE BLOWPIPE AND ITS USE IN CHEMISTRY AND MINERALOGY.

THE blowpipe has for a long time been used by workers in metal for the purpose of soldering together small pieces of metal, by means of an easily fusible metallic alloy, corresponding to the nature of the metals in hand. It consists of a conical brass tube about two hundred and forty millim. long, bent at a right angle at the narrower end, but not with a sharp corner, so that with it the flame of an oil-lamp can be conveniently directed upon the piece of metal to be soldered, which is laid upon charcoal. The wide end of the blowpipe is placed between the lips, the lower end directed toward the flame, and a stream of air strong enough to produce the necessary heat is blown through it upon the flame. At the present time, however, this instrument forms one of the indispensable aids which the chemist employs in his laboratory, the mineralogist in testing and determining minerals, and the miner and smelter in examining ores and metallurgical products.

The blowpipe, as used in its simplest form by workers in metal, is subject to a serious inconvenience, since the moisture which unavoidably collects in the tube is finally driven out by the pressure of the air and produces disturbances in the flame. To avoid this, a hollow chamber is connected with the blow-pipe which serves to collect the moisture. Cronstedt attached a hollow ball somewhat below the middle of the tube, Bergman adopted a semicircular shape, while Gahn gave a cylindrical form to the part designed for retaining the moisture. Various other methods of improving and simplifying the instrument are more specially mentioned by Berzelius. The length of the blowpipe should be adapted to the owner's eye, so that the body to be treated may be held where it can be most distinctly seen. A shorter tube is therefore to be recommended for a near-sighted person, and a longer one for one who is far-sighted. The construction most used at present, proposed by Gahn and approved by Berzelius, is represented in one half the natural size in Fig. 1. The whole length from *A* to *B* is two hundred millim. The jet, *b*, so arranged that it can be removed, is most advantageously made of platinum, being either soldered together from not too thin a sheet, or turned from a solid piece. It is well to have two such jets; one with a smaller hole bored to a width of 0.4 millim., for qualitative assays only, while the other has a hole 0.5 millim. wide, and is used for such qualitative assays as require a strong flame, and for all quantitative assays. Experience has shown these to be the best dimensions. If the hole is too small it can be bored wider with a fine steel drill or broach, like that used by watchmakers. It must be bored from the inside toward the outside, and the projecting rim which is generally produced on the outside is then removed by using alternately the drill and a small file, until the hole appears perfectly round on looking through it with a magnifying-glass. Jets with too wide a bore are useful in but few cases. When, through long use, the platinum jet is covered with soot and the hole obstructed, it can be cleaned by removing it from the tube and heating it to a moderate redness on charcoal with the help of the blowpipe, or in the upper part of the flame of a spirit-lamp.

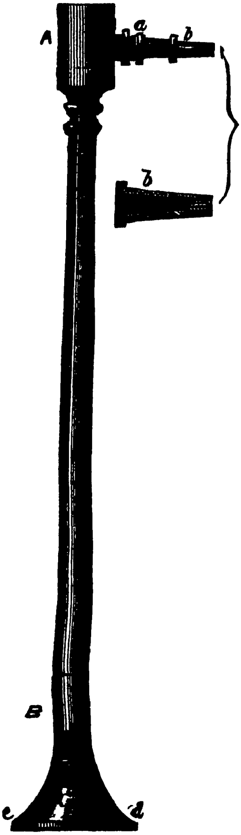

Fig. 1.

To avoid tiring the lip muscles by long blowing, Plattner has recommended the horn mouth-piece, *C*. By pressing this slightly opened mouth accustomed to it can blow uninterruptedly for a longer time and much more strongly than without it, nor is the least fatigue felt in the lip muscles. Particular care must be taken that the mouth-piece, *d e*, is properly curved, so that the rim of it may not cause unnecessary pressure on the mouth, and the diameter of the outer end should be about thirty-five millim. On account of the strong conducting power of silver, a blowpipe of this metal becomes so hot through long blowing, that it can scarcely be held with the naked fingers, and blowpipes are generally made of brass or German silver.

Mitscherlich has proposed a very convenient blowpipe, of somewhat different construction, for travelling, which is shown in Fig. 2. The cylindrical part, *A*, serving to retain the moisture, is attached to the long tube, which unscrews in the middle at *B*, while the smaller tube, *a b*, can be slipped into the half that is fastened to the moisture reservoir, and the other half, *C*, the mouth-piece of which, *D*, should be covered with silver when the whole is of brass or German silver, can be put over this like a case. The cylinder thus formed can be conveniently carried in the pocket.

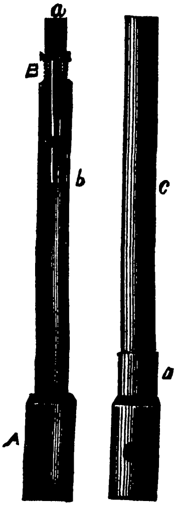

Fig. 2.

Many forms of mechanical blowpipes have been described, but they all fail in some essential point, except the one figured in the margin. This instrument perfectly fulfils all the conditions required: viz. throwing a perfectly steady oxidizing and reducing flame at will. The action will be readily understood by looking at Fig. 3. The indiarubber ball *C* is *gently* pressed by the hand or foot two or three distinct times, which fills the reservoir *B*. This reservoir must *not* be kept too full, otherwise it soon loses its elasticity, unless a very strong blast is required. The platinum

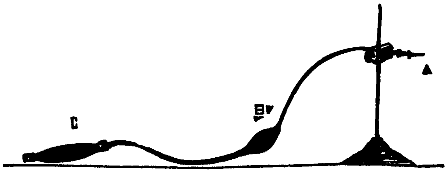

Fig. 3.

jet *A* must also have a smaller opening than the one used in the mouth blowpipe. In all other respects it is used in the same manner as the mouth blowpipe.

II. THE FUEL.

In case of necessity a candle flame may be used for many qualitative blowpipe assays, but when a stronger flame is required, as in quantitative assays, peculiarly constructed lamps must be used, in which rapeseed oil, olive oil, a mixture of alcohol and turpentine, or illuminating gas are burned. The flame of alcohol is poor in carbon and only suitable for a few blowpipe experiments. Rapeseed oil must be refined, as the unrefined oil smokes. Olive oil burns very well, but is sometimes bad because the blowpipe flame is surrounded by a broad yellow envelope, and is then useless for assays in which the colouring of the flame by the substance under examination is to be observed. The form of blowpipe lamp now used for rapeseed or olive oil is the same as was proposed by Berzelius. The cistern, Fig. 4, is of tinned sheet-iron, about one hundred and sixteen millim. long and coated with dark lacquer. The socket, *a*, is twelve millim. long in the clear, and five

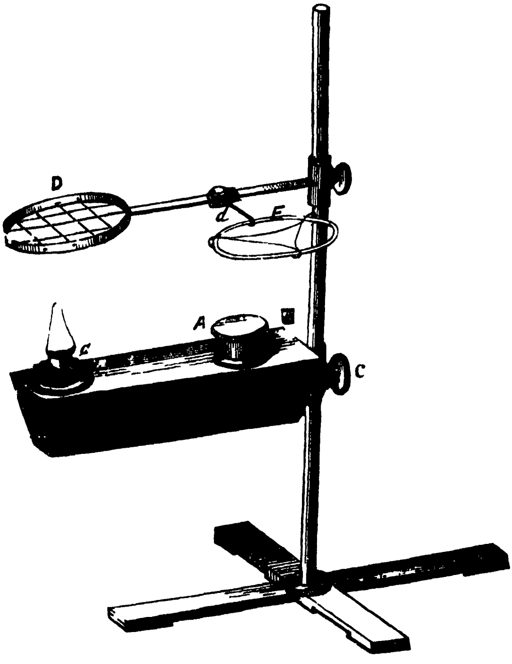

Fig 4.

millim. wide, and is filed slanting from right to left, so that the flame can be directed downward by the blowpipe when required. It is better, however, to effect this by having the lamp itself made with a side-movement at *e*, so that it can be bent over towards the charcoal. A cover can be screwed tight over the socket, and for this purpose is provided with a broad rim on which a leather washer soaked in wax is fastened with shellac. The oil is poured into a special opening, *A*, which can be likewise closed with a screwed cover. The wick is woven in a cylindrical form and is of cotton, like those used for Argand lamps. It is pressed out flat and folded lengthwise so as to come fourfold into the socket, to the width of which it must exactly correspond, fitting neither too loosely nor too tight, and its upper edge is cut parallel with the socket. The lamp itself is mounted on a brass stand and fixed on the brass rod by a screw, *c*. On the same stand is a brass ring, *D*, about fifty millim. in diameter, provided with a movable arm, and in it is a network of iron, or better still, platinum wire, which serves as a support for small porcelain vessels used in drying substances or heating fluids either over the free lamp flame or over the spirit-lamp. Since, however, this arrangement is not well suited for heating a small platinum crucible or a thin dish of platinum or porcelain to a red heat over a common spirit-lamp, there is a square hole in the movable part of the arm, *d*, into which the arm of an igniting ring, *E*, can be inserted. On the igniting ring is placed a triangle of platinum wire.

The introduction of illuminating gas in many chemical laboratories has rendered its use very convenient for the blowpipe experiments, and the burner proposed by Bunsen is best suited for this purpose. Through the neck, *g*, Fig. 5, which is connected with the gas-pipe by a rubber tube, the gas flows into the vertical tube, *a*, from below, issuing through a fine opening

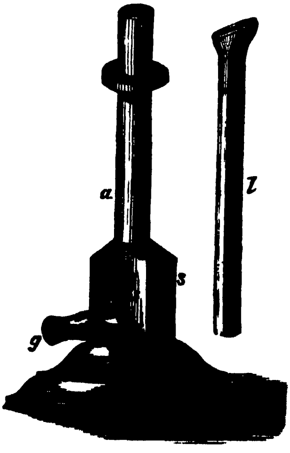

Fig. 5.

made by three slits meeting at a central point. Inside of the tube, *a*, the gas mingles with air entering through the side openings, *s*, so that if kindled at the upper end, it burns with a blue flame free from soot. While by this arrangement various experiments on the *colouring of the flame, the fusibility of substances, and with borax and salt of phosphorus* beads can be made, it may also be readily converted into a regular blowpipe lamp by slipping into *a* the small tube, *l*, so far as to cut off the access of air through *s*. The tube, *l*, is inclined at the top like the lamp socket, and has a slit ten to eleven millim. long and about 1.5 millim. wide. When the air is thus cut off, the gas burns at the upper opening with an illuminating flame, which may be made as large as an ordinary lamp flame by regulating the access of the gas.

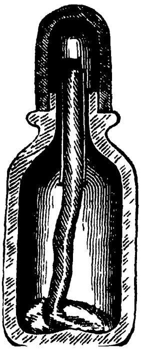
Fig. 6.

Besides the lamp for oil, etc., above described, a simple spirit-lamp, Fig. 6, is employed with advantage for examining many substances for volatile ingredients, in small matrasses and thin glass tubes, and for fusing various substances with bisulphate of potassa in a small platinum spoon, as well as for ignitions, etc. A larger spirit-lamp can be employed at home than on a journey, where the apparatus must be as compactly arranged as possible.

III. THE BLAST AND THE FLAME.

The blast with the blowpipe is not produced by the respiratory organs, because then it could not be long sustained, and an unbroken current of air could only be kept up for a short time; but it is produced by the muscles of the cheeks. The mouth is filled with air, which is forced through the blowpipe by these muscles, and while blowing, the connection between the chest and the cavity of the mouth is closed by the palate, which acts at the same time as a valve, so long as the mouth is sufficiently full of air, and respiration is effected only through the nose. When, however, the tension of the cheek muscles decreases, air is again admitted into the mouth through the throat during the act of expiration, and the cheeks thus inflated anew without interrupting the blast.

Beginners generally commit the error of not closing the connection between the chest and the mouth at the right time, thereby allowing the lungs to work directly for a longer or shorter period. That *this* style of blowing may be injurious to the health is not to be doubted. Persons unaccustomed to the use of the blowpipe can learn to produce a steady stream of air by taking care to breathe neither too fast nor too slowly, but just as usual, and in a distinctly audible way during the blast, and to continue this audible breathing until they can produce an uninterrupted stream of air of uniform force without the least straining. Success is not indeed immediate, but a few days' practice will cause improvement, and after a while such facility is attained that no further special attention need be bestowed upon the blast itself, and any fear of injury to the health is entirely dissipated.

It is impossible to prescribe the manner of holding the blowpipe while blowing, and the position of both forearms, while treating an assay, since this depends upon habit; but the blowpipe can be held very securely and conveniently by taking the long tube between the fingers of the right hand, but so that the inner joints of the index and middle fingers are above and the inner joints of the other two fingers are below the tube, while the thumb is extended and supports the tube with the end joint, where the mouth-piece is attached. It is soon found that the position of the forearms is more convenient when they only rest against the edge of the table than when the elbows are placed upon it.

After learning to blow a strong unbroken current through the blowpipe, there is no difficulty in producing a good flame by conducting the current through the flame of a lamp, but in addition to this a knowledge of the flame and its separate parts is necessary. On looking at the flame of the blowpipe lamp it will be observed, if the wick is not drawn out so far that it smokes, that it is composed of four separate parts. If a burning taper is placed beside it, the same parts may be even more distinctly observed in its flame.

Fig. 7 represents a candle flame, at the base of which is seen a small light-blue part, *a b*, surrounding the flame at this point, but narrowing as it recedes from the wick and entirely disappearing where the sides of the flame ascend vertically. In the middle of the flame is a dark cone, *c*, surrounded by the illuminating flame proper, the mantle, *d*, on the outer edge of which is a very thin, scarcely visible envelope, or veil, *a e b*, which widens toward the tip of the flame and is the hottest of its several parts. On holding a rather fine platinum or iron wire across the flame at *f f*, it is seen to swell most and to become white-hot in the envelope, *a e b*, while in the darker portion, *c*, it scarcely glows. The cause of this is as follows: the heat of the flame radiates back upon the tallow, wax, etc., and melts these substances, which are then sucked up through the capillary force of the porous wick and brought into a temperature high enough to convert them into vapour. While these heated vapours are ascending, the air enters from all sides and its oxygen effects the combustion; but this takes place only on the outer limit of the flame, forming the envelope, *a e b*, which consists of carbonic acid and steam, and here too the flame is hottest. In consequence of this high temperature the vapours behind this envelope, consisting chiefly of the two kinds of carburetted hydrogen, separate into their constituent parts, and the separated carbon is made to glow, causing the light of the flame and the existence of the part *d*. As the free carbon approaches the veil, which is rich in oxygen, it is burned to carbonic oxide, then to carbonic acid. The dark cone in the flame consists of undecomposed vapours, since the heat of the veil decreases below and toward the middle of the flame. The air having access to the flame from all sides, at *a b*, produces a very perfect combustion, resulting in the light-blue portion; but as there is not enough oxygen to convert the carbon into carbonic acid, only carbonic oxide is formed, and this causes the blue colour. Of these four parts three can be as easily distinguished in the flame of the oil-lamp as in that of a taper, but the fourth, scarcely illuminating portion, is only to be per-

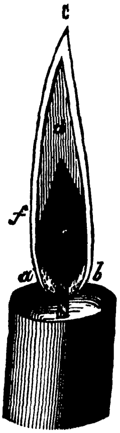

Fig. 7.

ceived imperfectly and by careful observation. Only two of the parts are generally employed in blowpipe assays; the slightly illuminating envelope for oxidation, and the illuminating portion for reduction. With the blowpipe, each of these parts may be made to work by itself, and we may therefore call the slightly illuminating envelope the *outer* or *oxidizing flame*, and the illuminating part the *inner* or *reducing flame*. Cases occur, however, where the slightly luminous flame oxidizes too strongly and the luminous flames reduces too strongly, in case a lively flame is needed. Under such circumstances the blue part of the flame is best employed. The manner in which the different parts of the flame can be rendered effective with the blowpipe will be particularly described in the following pages.

I. THE OXIDIZING FLAME.*

On blowing into the lamp flame from one of its narrow sides, so that the jet of the blowpipe extends about to the third part of the breadth of the socket, and the current of air, almost touching the wick, passes directly through the middle, a long blue flame, *a b*, is produced, which is really the same as *a b* in Fig. 7, at the base of the free flame, except that it appears in another form, and contains all the burning gases which are developed; it here forms a slender cone, while there it only incloses the lower part of the flame. The hottest point is in front of the tip of this flame, where the most perfect combustion of the developed gases occurs. This hottest portion forms an envelope about the whole of the free flame, but here it is rather contracted to a point in front of the blue flame, surrounding the point of the latter with a cone of flame, which also extends to some length from *a* to *c*, and has a pale-blue colour. As before mentioned, the hottest point is just in

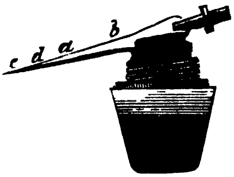

Fig 8

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* Throughout the rest of this work the oxidizing flame will be designated by the letters O. F.

front of the tip of the blue flame at *d*, but it rapidly decreases in temperature toward *c*, and still more rapidly toward *b*. When a very high temperatnre is not required, the oxidation is best effected by heating the assay as far from the point of the blue flame as will admit of producing the necessary temperature.

An essential condition in producing a pure O.F. is a wick free from charred threads and hardened particles, and cut parallel with the slanting edge of the socket, since otherwise yellow streaks easily occur in the blue cone of flame, which are rich in carboniferous particles, and have a reducing effect on the assay. When an assay is treated on coal, a very strong blast should not be used, or else a part of the coal will be burned to carbonic oxide gas, which counteracts the oxidation.

Molybdic acid affords the best material for practice in producing a pure O. F., as it immediately gives a brown glass with borax in an impure O. F. A moderate amount of it is dissolved in borax on a platinum wire in the O. F., at three to four millim. distance from the point of the blue flame, and yields a clear yellow glass, colourless when cool. On treating this glass directly with the tip of the blue flame for a short time, it becomes brown, and after blowing longer quite opaque, because the molybdic acid is brought with extraordinary ease to a lower state of oxidation, viz., binoxide of molybdenum. Even a yellow streak in the O. F. produces a brown colour in the glass. The sooner a borax bead quite opaque with binoxide of molybdenum can be rendered clear again, so much the purer is the O. F. employed, provided it is effective enough. To be certain that a sufficiently strong O. F. can be produced, it is only necessary to try to fuse the end of a platinum wire, 0.1 millim. thick to a globule. One end of the wire is bent at a right angle and held in the O. F. with its axis corresponding exactly with the axis of the flame and so that it does not vibrate. With a pure and strong flame a globule will soon be observed to form suddenly, being larger in size the stronger the flame employed.

2. THE REDUCING FLAME.*

On blowing from the narrow side directly into the middle of the flame, so that the blowpipe tip reaches very little or not at all into it, and the current of air, Fig. 9, passes at a somewhat greater distance above the wick than in Fig. 8, the whole of the flame assumes the same direction as the stream of air, and appears as a long luminous cone, *a b*, the end of which, *a*, is surrounded by the same pale bluish flame which can be observed with some care in the free flame, but which here extends to *c*. By thus blowing into the flame, the gases arising from the wick are burned, and the carbon, separating in infinite particles, is rendered white-hot, and then, likewise consumed, producing, in common with the vapour of water already formed, the outer flame, which is plainly visible as far as *c*. A small part only of the dark cone is still visible, immediately above the wick, and reaching to *d*, and between *a* and *d*, but somewhat nearer *a*, is the most active part of the flame. If this is directed, for example, upon a reducible metallic oxide, so as to completely surround it and cut off the access of air, the oxide will, owing to the tendency of the free carbon in the flame to take up oxygen, either be entirely or partly freed from its oxygen, according as the oxide is easy or difficult to reduce, and the metal hard or easy to fuse, or according as the reduction is effected on coal or in a solution in glass fluxes on platinum wire.

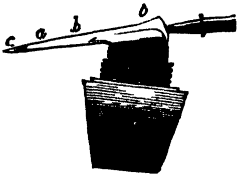

Fig 9.

A good R. F. is harder to produce than a good O. F., and especial care must be taken that the assay be brought only into the most active part of the flame, and *completely enveloped by it*, while the flame must be kept thus unchanged for a long time.

Binoxide of manganese and the oxides of copper and nickel will serve for practice. Binoxide of manganese dissolved in a borax bead on platinum wire in the O. F., gives an amethyst-red glass, or when used in excess a black opaque bead, and the sooner the binoxide can be reduced to protoxide, and the bead thereby rendered almost colourless, so much the more perfect is the R. F. employed. A similar bead made with oxide of copper or nickel, and then shaken off, and treated on coal with the R. F., will soon prove whether the flame produced has the *right effect or not. Both of these oxides can be reduced to the metallic state, the copper uniting to a small button, while the nickel comes to the sides of the glass in a state of cohesion.* The sooner the glass becomes clear and colourless, the purer and more powerful is the R. F.

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* The letters R. F. will be used hereafter to designate the reducing flame.

IV. THE SUPPORT.

In treating an assay with the blowpipe flame, it must be supported on a body which, during the heating and fusion of the assay, will neither combine with it, nor cause wrong results in case the support is combustible. In many cases the assay is laid directly on such a body, but in many others this occurs indirectly, and the support is therefore either a direct or an indirect one.

a. THE DIRECT SUPPORT.

1. **Coal.** Well-burned charcoal is especially suited for the support, as it helps to increase the heat when necessary. It is best made from mature light woods, as the pine and alder, and cut into parallelopipedons, eighty to one hundred millim. long, and into square prisms, according to the assays for which they are intended. Only those sides of the coal which show the edges of the annual rings are used. Good charcoal for blowpipe assays cannot, however, be found everywhere, nor is it always possible to produce sufficiently firm coal by charring perfectly dry wood in vessels, and it is therefore advantageous, particularly for quantitative assays, to make coals of the requisite shape out of not too fine coal-dust with some binding material. Starch-paste, which Plattner has recommended as the best binding material, is prepared from one part by weight of starch-meal and six parts of water. The starch is

stirred to a thin paste in an earthen vessel with a little of the weighed or measured water, and the rest of the water is poured, boiling hot, upon the paste, and the whole briskly stirred with a beater, until all the meal is converted into paste. To prepare blowpipe coals, this paste is rubbed in a porcelain mortar with successive additions of charcoal dust, until the mass in the mortar becomes too tough for any further admixture of coal dust. Enough of coal dust is then kneaded in with the hands to render the whole mass stiff and plastic, and it is then worked thoroughly. From this mass various forms of blowpipe coals can be made, as will be hereafter described. When made they are allowed to dry gradually and thoroughly, and are then heated to a low redness in a covered crucible, so as to char the binding material. Small pieces can be ignited in a covered porcelain crucible, over a spirit-lamp with double draught or a gas-lamp; when preparing larger pieces, or a sufficient supply of the coals, it is best to choose a spacious crucible of clay, or stout sheet-iron, which is covered with a close-fitting cover, and heated in a small wind-furnace with a very weak draught, between glowing charcoal, or in some other moderately strong fire. The charring is complete when combustible gases cease to issue from beneath the cover, and when, on raising the cover, the coals in the upper part of the crucible are perceived to be at a low red heat; the crucible is then removed from the fire and allowed to cool with the cover on. The coals are of the proper firmness and ring like ordinary good charcoal when thrown on the table.

The various forms of coal to be prepared, are as follows: for assays where no regard is had to the coat, and for refining copper, small coals of a flat dish-like shape are made. In producing them, the mould to be hereafter described for the clay capsules, Fig. 22, is used, and only a special stamp is needed, which is best made of boxwood; the moulding part has the form of the segment of a sphere, constructed with a radius of twenty millim., Fig. 10. After strewing the mould, *A*, Fig. 22, with coal dust, a strip of paper about fifty millim. long and five millim. wide is laid over it, the cavity filled with the prepared mass, and this pressed together with the stamp, Fig. 10, which has been dipped in coal dust. By means of the two projecting ends of the paper, the dish-shaped coal is lifted from the mould, and then set aside to dry in a moderately warm place, after which it is ignited as before mentioned. Fig. 11 shows such a coal.

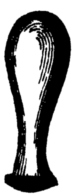


Fig. 10.

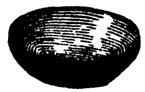


Fig. 11.

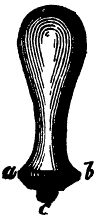


Fig. 12.

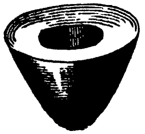


Fig. 13.

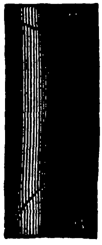


Fig. 14.

Small-coal crucibles are very well adapted for decomposing compounds of silicic acid as well as for quantitative assays. The crucible mould, Fig. 24, to be hereafter described, is employed in making them, and the metallic plug is replaced by a wooden one, Fig. 12, the diameter of which at *a b* is twenty-seven millim. and at *c* nine millim. The iron mould is first pressed full of the mass, which has previously been rolled into a ball and dipped in charcoal dust, and then the wooden plug, Fig. 12, is dipped in coal dust and set upon it, so that the part *c* comes exactly in the middle of the mass, which is then pressed together. After removing the plug by turning it gently, the mould is taken apart in the way to be described in making clay crucibles, and after cutting off the projecting edges on the two opposite sides, the coal is so far ready that it requires only to be dried and ignited in a closed vessel. The coal crucible in the natural size is shown in Fig. 13. The depth of the cavity in such a crucible need only be six millim. and when a deeper hole is necessary for any quantitative assay it can be bored out and widened as required, with the help of the coal-borer to be described hereafter. In order to facilitate the handling of these coals, capsules, and crucibles, a cylinder, Fig. 14, is used as a support for them, which is sixty to sixty-five millim. high and twenty-five millim. in diameter, being made of any mass which is easily worked, fusible with difficulty or not at all, and is a poor conductor of heat. It is provided at each end, *A*, *B*, with cavities corresponding to the size of the coal to be supported. Pumicestone or porous burnt fire-clay is a suitable material. To prepare a cylinder from fire-clay, the dry powdered clay is well mixed with an equal volume of coarsely beaten charcoal and then made plastic with water. The cylinders formed from this, either by hand or with the aid of a special mould, are allowed to dry thoroughly and then ignited in a loosely-covered crucible among coals.

In quantitative blowpipe assays when roasting ores in clay capsules, and when fusing lead, bismuth, tin, and many nickel and cobalt assays in clay crucibles, a hollow coal is required, which is secured in an especial coal-holder, and the cavity of which must be covered with some suitable coal, also hollowed out, when a fusion is to be performed. For these coals a mould of hard wood can be used, constructed as follows: the main part of the mould, *C*, Fig. 15, consists of four pieces, which fit each other exactly and are held together by a brass ring, which can be drawn more or less tight by the screw *g*. These four pieces surround a prismatic space forty millim. high and thirty-five millim. square. *A* and *B* are stamps, the rims of which, *a b* and *c d*, have a diameter just equal to the distance between two of the strong brass pins fastened opposite one another vertically in the four pieces of the mould *C*, and which serve to bring the stamps *A* and *B* exactly in the middle of *C*. The projecting portion, *e*, of the stamp *A* is eighteen millim. long and has a diameter of twenty-two millim. at the top; the part, *f*, attached to *B* at *c d*, forms a segment of a sphere which has a breadth of twenty-two millim. at its

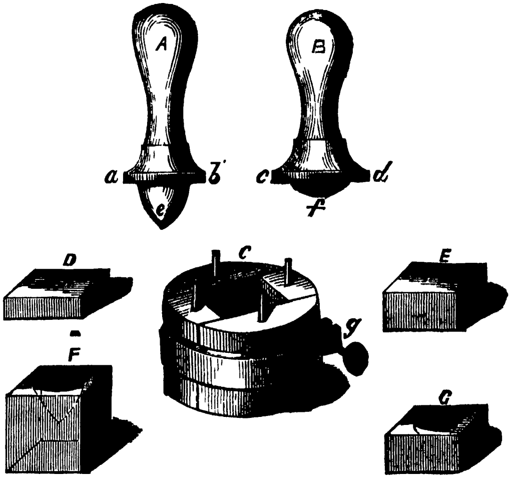
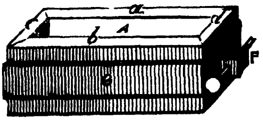
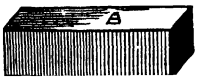


Fig. 15.

junction with the rim, and is nine millim. thick. *D* is a prism fitting exactly into *C*, and serving as a bottom in making coals for roasting and fusions. *E* is a prism seventeen millim. high, which is laid upon *D* when only coal covers are to be made, and hence also serves as a bottom. When coals are to be made in this mould, a piece of paper corresponding to the size of the mould is laid on the bottom prism, the side surfaces are rubbed with a little coal dust, the empty space pressed full of the coal mass, and the requisite stamp, the moulding part of which has likewise been dipped in coal dust, is pressed firmly into it, turning the stamp a little at the same time on its axis and then drawing it carefully out. By loosening the ring and removing it from the mould the separate pieces can be easily taken away by sliding each one downward from the moulded coal, which is then ready for drying and charring the binding material. Such coals must be dried with care, as they are liable to crack if put immediately in a very warm place. *F* is a coal as it is used, when secured in the coal-holder, for roasting in clay capsules and fusions in clay crucibles, and *G* is a coal which serves to cover the cavity in the former coal during fusions. Both coals are bored through shortly before use, the former on the side, the latter in the middle of the hollow, as will be described in the proper place; the cavity in *F* can also be made deeper and wider with a coal-borer, as required.

When there is a total lack of charcoal suitable for qualitative assays, in which long pieces in the shape of a parallelopipedon are generally needed, they can also be made in the above manner, by means of the mould shown in Fig. 16. In this case, however, if the coal dust on burning leaves a considerable amount of ash, it must first be purified by digesting it in aqua regia and then washing it well with hot water. The coals, about eighty millim. long, twenty millim. wide, and from ten to twenty millim. thick, are made in the following way: the main part, *A*, of the mould, which consists





of four pieces, *a b c d*, held together by a brass band, *E*, and surrounding a space, eighty millim. long, twenty-one millim. wide, and thirty millim. high, is placed upon a firm, even support, and a piece of wood five millim. thick, corresponding in length and width to the inside of the mould, is laid in it for a bottom. This bottom is covered with a piece of paper of the same size, the empty space filled with as much of the coal mass as is needed to make a coal of a given thickness, and on this is placed another piece of paper, the size of the first, and finally the moulding part, *B*, having a section twenty-one millim. square, is inserted and the mass pressed together. This dône, the screw, *f*, is turned enough to loosen the brass band, when the four pieces, *a b c d*, are drawn out separately, and the coal, after being freed from the adhering papers, is ready to be dried and charred. By holding the mould in both hands and pressing down the piece *B* with the thumbs, the moulded coal may be removed without taking the mould apart, but its interior surfaces must be wiped off every time before moulding a new coal, lest particles of the mass should adhere to them, and the easy separation is promoted by rubbing the sides of *A* with a little coal dust. The binding material of these coals must likewise be charred when they are perfectly dried. These long coals, as well as cut pieces of charcoal, after being used, are best cleansed from the coats, &c., which may be on them, and prepared for further use, by means of a file or rasp.

2. **Platinum** in the shape of wire, foil, and spoons.—The best platinum wire for qualitative use is about 0.4 millim. thick, and is cut into pieces about forty-five millim. long, with a loop at one end, Fig. 17, *A*. This serves as a support for borax and salt of phosphorus beads, which can thus be very conveniently examined, and are quite free from the false play of colours that often appears on coal, through the position of the bead on the black support. In examining metallic alloys, however, and in reduction assays where easily fusible metals separate, platinum wire cannot be used, but coal must always be used as a support. When in use the wire is either fixed in a soft cork or secured in an especial holder, Fig. 17, *B*, which also serves as a case for several wires. To prevent

injury to the wires from the screw, holders are used in which the wire is inserted into the middle of two slits crossing each other at right angles; the latter are then shut tight by a band which is thrust over them and arranged to screw up and thus hold the wire. In Fig. 18, the upper part of such a holder, with the band, *a*, is represented in the natural size. The loop is most readily cleansed from adhering substance by warming it in a test tube with dilute hydrochloric acid, and then rinsing it with distilled water. Besides several slender wires there may be another, 0.6 millim. thick, and likewise bent to a loop at one end, Fig. 17, *C*, which is of advantage in testing for tantalic and tungstic acids, etc., where the substance must be fused with alkaline carbonates. It is either held with the fingers or fastened in a small cork.

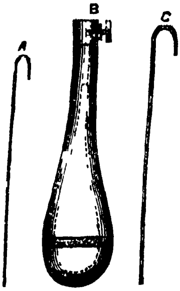


Fig. 17.

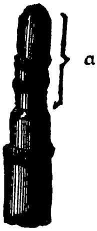


Fig. 18.

The use of platinum foil in qualitative examinations is very limited. The thin rolled foil is cut into strips about sixty millim. long and fifteen millim. wide, and when in use the free end is either held in the forceps or thrust into the end of a long piece of charcoal, between the yearly rings. Metallic substances in the reguline state, or such as are easily reduced and fused during the blast, must not be treated on platinum foil, since they combine with it and render the corresponding spot useless. The foil is generally used to fuse substances containing manganese with carbonate of soda, which becomes bluish-green on cooling, from the presence of manganate of soda, and thus the presence of manganese is indicated.

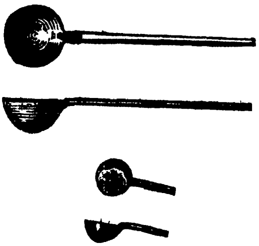


Fig. 19.

A platinum spoon is necessary for many assays, and it is, in fact, advantageous to have two, one about fifteen millim. in diameter, and a smaller one, Fig. 19, about nine millim. in diameter. On using the larger spoon, the handle, which must also be of platinum, is inserted into a small wooden holder, or into a piece of cork; the smaller spoon is held fast by the handle with the forceps. The larger spoon is used for fusing certain substances with bisulphate of potassa, for heating the gold obtained from a quantitative assay, and for other purposes; the smaller one, on the other hand, serves only for the fusion of certain substances with saltpetre. If it happens, after a fusion with saltpetre, that the spoon does not become clean by dissolving the fused mass in water, on account of adherent particles of metallic oxides, it is only necessary to melt a little bisulphate of potassa in it over the spirit-lamp, and then to cleanse it with water.

3. **Glass Tubes and Matrasses.**—For recognizing in minerals, ores, and products, substances which become volatile at a high temperature by access of air, tubes recommended by Berzelius are used, from one hundred and twenty to two hundred millim. long and about six millim. in diameter, which are open at both ends. The assay is placed near one end, which is then inclined downward, while the other end is warmed over a spirit-lamp, so as to create a draught through the tube, and then the spot where the assay lies is to be heated. When but little heat is required for driving off the volatile substances, or those which become volatile, the free flame of the spirit-lamp is used; but if otherwise the blowpipe flame must be employed. The tube is inclined more or less according to the strength of the draught desired. The volatile substances formed during the roasting either pass off as gases, or are sublimed upon the interior of the tube, and can thus be easily recognized. A small supply of these tubes is to be kept, and when one has been employed it is broken off by filing a notch above the spot used and then cleansed and kept for another assay. When it finally becomes too short, one end is closed by melting, and it will still serve for a sublimation test. To prevent the assay from falling out of the inclined tube before it adheres to the glass, Berzelius recommends bending one end of the tube at an obtuse angle, Fig. 20. The assay is then laid in the angle *a*, and the tube inclined as required.

Fig 20.

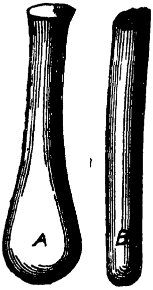


Fig. 21.

A matrass is a tube closed below and blown into a bulb, Fig. 21, *A*, and is sixty to seventy millim. high. It is used for ascertaining the presence of *water or any volatile body in a substance, or in case a substance which decrepitates strongly is under treatment and is to be further examined. The matrass is heated over* a spirit-lamp, and when used for another assay *must be cleansed with dilute acid or water and* thoroughly dried, which is accomplished very simply and rapidly by warming it well over the spirit-lamp and sucking out the water in the form of vapour through a slender glass tube that reaches into the bulb of the matrass. Fresh air thus enters, and by continued suction removes every trace of water in the form of vapour. When combustible bodies, like sulphur, arsenic, etc., are to be sublimed from a mineral, ore, or product, a glass tube, Fig. 21, *B*, five to six millim. wide and seventy to eighty millim. long, is used, which is melted together at one end, but is not enlarged, so that neither combustion nor partial oxidation of the combustible bodies can take place, as would be caused by a slight current of air. A small supply of matrasses and closed tubes should also be kept on hand.

4. **Capsules and Crucibles of fire-clay.**—The capsules are used for roasting minerals, ores, and metallurgical products, which are to be quantitatively examined for the metal in them, as well as for roasting substances consisting of a mixture of earthy parts with metallic arsenides and sulphides, which are to be examined only qualitatively for the earths or metals, as for example, ores dressed on a large scale. These capsules are made in the following way: First a stiff paste is made of elutriated fire-clay. Then the moulding surfaces of the boxwood mould, Fig. 22, *A B*, of which *A* has a width of 20.5 millim., above, and a depth of 7 millim., while *B* is constructed on a radius 0.8 millim. smaller, are rubbed with oil and a strip of thin paper, fifty millim. long and five millim. wide, is laid

over the cavity of the mould. A ball of the soft clay mass, about twelve millim. in diameter, is then placed on the middle of the paper and pressed firmly into the cavity with the fingers, and after placing *A* on a firm level support and holding it with one hand, the convex part *B*, is pressed with the other hand in a vertical position directly into the middle of the clay as far as necessary, being turned a quarter of a revolution on its axis. By this means the superfluous clay is pressed out at the side, and *B* can be easily drawn out by turning it carefully. As much of the clay which has been forced out is cut off as is necessary, and then the edge of the capsule is examined, to see whether it is sufficiently thin all around, or whether one side is thicker than the other. Fig. 23 shows a section of such a capsule, in the natural size, which must be only 0.8 millim. thick before it is burned. If it is too thick on one side, *B* must be again pressed into it, rather more on the thick side, or exactly in the middle if it is too thick all around. After carefully withdrawing the convex piece, *B*, and cutting away the superfluous clay, one end of the paper strip is taken in one hand and the other end in the other hand, and the capsule cautiously lifted from the mould. When, through want of practice, the capsules become disturbed in removing them from the mould, they can be restored to shape by pressing them separately with the fingers against the convex piece, *B*, on all sides, before they have become dry in the air. During this operation the paper strips separate of themselves, and the basins are set aside to dry in a warm place, after which they are put in a vessel of baked clay, which is set uncovered in a potter's baking furnace, or in some other fire, where they can be brought to a red heat, as in an assay muffle, which has just been fired up, or in a simple coal fire. They may also be baked in a platinum crucible over a spirit-lamp with a double draught, or a gas-lamp. These basins shrink a little in baking, but remain just the size required.

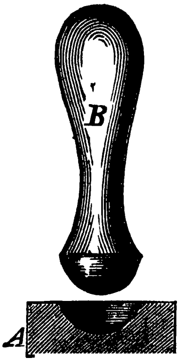


Fig 22.

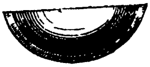


Fig. 23.

The crucibles are used for quantitative assays. They are formed in a brass mould, consisting of a plug and a box, the latter being composed of two parts held firmly by a ring. Fig. 24 represents this instrument, and Fig. 25 a prepared clay crucible. *A* is the plug, with four conical openings at *a* to let out the excess of clay put into the mould. The moulding part has a diameter of nineteen millim. above, and is fourteen millim. long; *B* is the box, consisting of two halves, fitting exactly together and forming a blunt cone. At *b*, on the inner side of each half, which is about 0·8 millim. from the plug all round, the corners are somewhat blunt, so that slight cavities are formed in the box at two opposite points, and when moulding crucibles these become filled with clay, thus preventing the crucible from turning when the plug is turned. *C* is the binding ring into which the box is slipped, so that it can be easily lifted out, and the lower surface of the box and ring are in the same plane.

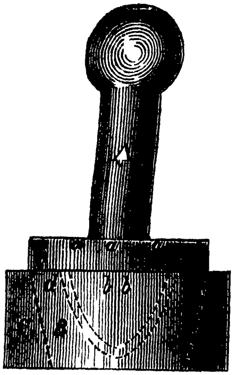


Fig. 24.

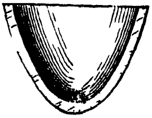


Fig 25

To mould a crucible, small balls are formed with the fingers from a stiff paste of water and elutriated fire-clay, each containing rather more than is needed for a crucible, and they are then allowed to dry in the open air, until they can only be pressed between the fingers with difficulty. The moulding surface of the box and plug, and those faces which are to lie upon one another, are then rubbed with a very little oil, and the mould, with the ring, is placed upon the anvil, which rests upon some elastic support, such as a woollen cloth folded several times. The clay ball is then put into the box and the plug driven in so far, in a vertical position, with a wooden mallet, that the projecting rim, *c*, rests upon the edge, *d*, of the box. The plug is then lifted out by turning it, which also removes the superfluous clay; the box is pressed out of the ring from below, and while one half is held between the fingers of one hand and the other with the other hand, the halves are separated in succession from the moulded crucible. This is best done by sliding one half down a little loosened on the other half so as to loosen the crucible, and then the first half is pressed gently against it while the other half is in the same manner and entirely detached, when the crucible can be removed in a perfect state from the first half. The crucibles, after being freed with a sharp knife from the two projecting bits of clay, are set aside to dry, either at first in the fresh air, or else directly in a warm place, and are then baked in the same way as the clay capsules.

No time should be spared in making clay capsules, since it is an essential condition that they should be quite thin, and the clay must not be worked up too hard or too soft. If too hard the capsule is moulded with difficulty, if too soft it can seldom be lifted from the mould without tearing. The consistence proper for the clay mass is, however, very soon learned. When the mould is new, the oil rubbed on the surfaces generally soaks into it, and the moulded basin cannot be taken out without tearing it, and it is well when using a new mould to rub it several times with oil and let it soak in thoroughly. *A* must likewise be rubbed with less oil than *B*, since otherwise the basin may easily adhere to *B*, and be lifted out with it. It is far better to buy coal capsules and clay crucibles in quantities of 500 or so at a time. They are much better made, and very nearly as cheap as making them oneself.

5. **Bone-ash.**—It is used to make small cupels upon which auriferous and argentiferous lead obtained from blowpipe assays is cupelled. Two grades are used, the *sifted* and the *elutriated* bone-ash.

Bones of quadrupeds are thoroughly calcined, the perfectly white portions, free from coaly parts, are selected, broken up and stamped in a mortar until the powder will go through a fine hair sieve. This yields the sifted bone-ash. A portion of the sifted bone-ash is put into a large beaker glass, which is then nearly filled with pure water, and the whole stirred with a glass rod and afterward allowed to stand a moment. During this time the coarse particles settle, while the finer ones remain for the most part suspended in the cloudy water, which is then carefully decanted into another beaker and allowed to remain quiet until the fine particles have settled, when the most of the water is poured off. As some fine particles settle with the coarse, the elutriation should be repeated until the water is only slightly cloudy. The fine elutriated bone-ash is then brought upon a filter, so that most of the water flows off, and is then dried and heated to redness. Both sorts readily absorb moisture and must be kept in glass-stopped bottles. The coarse powder remaining from the elutriation can be again pulverized and elutriated. The manner of making the cupels will be given in the description of the cupel moulds, under instruments.

b. The Indirect Supports.

1. **Soda-paper.**—In the quantitative determination of several metals the weighed and prepared assay must be wrapped in something which withstands the first action of the blowpipe flame, so as to prevent the particles of ore from being blown away. Harkort found fine letter-paper, soaked in a solution of carbonate of soda and dried, to be most suitable, and this paper may be used with advantage for wrapping up bulky charges in qualitative analyses also; but since letter-paper frequently contains foreign substances, such as oxide of cobalt, fine filter-paper should then be substituted for it. Thin strips of both sorts of paper are drawn through a solution of half an ounce of crystallized carbonate of soda, free from sulphate, in an ounce of pure water, which is put in a shallow vessel, such as a porcelain dish. The strips are dried slowly in the air, or in a moderately warm place, and are then cut into pieces thirty-five millim. long and twenty-five millim. wide, and kept for use. When used they are made into small cylinders, as will be described hereafter.

2. **A mixture of seven parts charcoal and one part fire-clay.**—It is used to line the small clay crucibles in quantitative tin and lead assays, and is made thus: Seven parts of very fine dry charcoal powder and one part of elutriated fire-clay are weighed out, the latter is thoroughly mixed with water in a

shallow dish, and the coal dust is then poured in and kneaded with the clay water to a paste, which is allowed to dry in a warm place, and afterward rubbed to powder again, in which state it is kept for use. To line a clay crucible with this mixture, a small quantity is made into a paste with water in a small porcelain dish, and part of it rubbed inside of the crucible, so as to lie about three millim. thick at the bottom and thinner on the sides, especially about the edge, as is seen in Fig. 26. While part of the water soaks immediately into the baked crucible, another portion remains in the paste, which is still so soft that it may be rubbed smooth on all parts, with the dry plug of the crucible mould, Fig. 24, *A*. The lined crucible is then thoroughly dried over the free lamp flame.

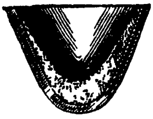


Fig. 26.

V. INSTRUMENTS, SMALL VESSELS, AND OTHER OBJECTS USED IN BLOWPIPE ANALYSES.

1. **A delicate Balance.**—For quantitative assays this must be capable of indicating with precision an additional 0.1 milligr. when loaded with two decigr., and should be so made that it can be easily set up and taken apart. Fig. 27 is a perspective view of such a balance, as constructed by A. Lingke, for blowpipe assays. The beam, which is one hundred and eighty millim. long, moves on carnelian plates, and all of the brass work is gilded. The tongue from *a* to *b* is one hundred millim. long, and the cords, including the hooks, one hundred and forty millim. The scale-pans attached to the cords are thirty-three millim. in diameter, and very slightly concave, and on each of them stands a small gilt pan fifteen millim. wide and four millim. deep, to receive the objects to be weighed and the weights. The two larger pans, *g*, *g*, each twenty millim. in diameter, are used for weighing bulky substances. The balance is set up on a low box, inside of which it can be packed, together with other instruments, when taken apart. On the lid of the box is screwed a stout upright brass rod, to which the balance is secured by a screw, and the beam is raised by a fine silk cord which passes over three pulleys,

c, *d*, *e*, the lowest one, *e*, being separately screwed in. This cord is attached at one end to the support of the beam, and at the other end to a knob, which is fastened in the box, and can be turned so as to wind up the cord. The brush, *f*, fastened on a movable brass arm, serves to prevent unnecessary swinging of the tongue. When the balance is to be used for determining the specific gravity of minerals, metallurgical products, &c., the necessary pans can be made for it. It is very advantageous to protect the balance against dust and currents of air by a glass case. Lingke has constructed one for this purpose, which can be folded up and transported on a journey.

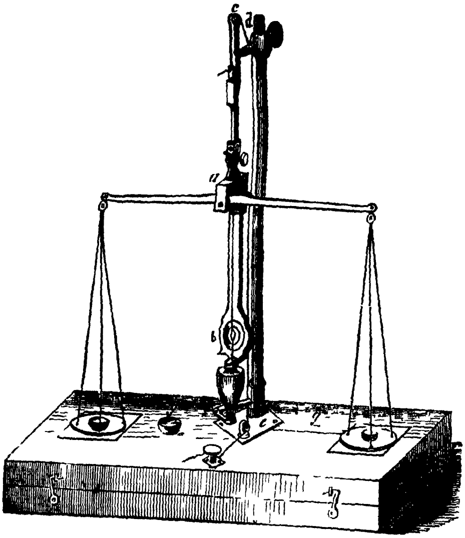


Fig. 27.

2. **Weights.**—The fittest weight for blowpipe assays is the gramme used by Harkort. One decigramme = 100 milligr. serves as the assay centner (hundredweight), and is the 37.5 part of the assay centner used at the Freiberg Smelting Works, which contains exactly 3.75 grm.

The highest weight required for blowpipe assays is a one-hundred milligrm. piece, but sometimes a greater weight is very desirable, and we can always attain our object by having a set of weights, which is best made of silver, and consists of the separate pieces given below. The fractions of a milligramme are made of pith. On each piece the weight in milligrammes is given, the figures from 1000 to 100 being engraved, while on the other pieces down to one milligramme, which are quite thin, they are struck or pressed, and the fractions of a milligramme are only distinguished by their size.

The following is a list of the pieces composing a set of blowpipe weights for the Freiberg balances:—1 piece of 1 grm. in weight, 1 piece of 5 decigrm., 2 of 2 decigrm., 1 of 1 decigrm. = 1 blowpipe assay centner = 100 milligrm., 1 piece of 5 centigr., 2 of 2 centigrm., 1 of 1 centigrm., 1 piece of 5 milligrm., 2 of 2 milligrm., 1 of 1 milligrm., 1 of 0.5 milligrm., 2 of 0.2 milligrm., 2 of 0.1 milligrm.

3. **Blowpipe Assay Scale, or Plattner's scale.**—The silver button obtained by a blowpipe assay of 100 milligrm. = 1 assay centner, of an ore poor in silver, is so small that its weight cannot be determined on the balance, and Harkort conceived the idea of measuring such buttons on a scale constructed for the purpose. This idea he executed so well that it is possible to determine with sufficient accuracy the proportion of silver in an ore, mineral, etc., even when it contains less than 0.0033 per cent. or 0.96 oz. troy in a ton of 2,000 lb.

Plattner, following Harkort's plan, prepared a scale, which is figured in the form made by Lingke in Fig. 28. The scale is made of ivory, and the lines, *a b*, *a c*, diverge 1 millim. at a distance of 156 millim. To determine the weight of a silver button, it is placed with a pair of fine forceps between the two convergent lines, *a b*, *a c*, and then, with

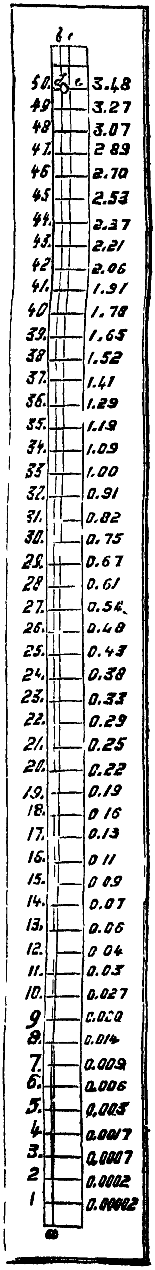


Fig. 28.

the aid of a magnifying-glass, moved into a position where the lines are just tangent to its sides, the eye being held vertically over it to avoid any parallax. On the left hand are the numbers of the transverse lines, and the figures on the right give the percentage of silver in an ore.

Should the button come about midway between two of the transverse lines, the percentage is found by simply dividing the sum of the percentages corresponding to these lines by two. Should it, however, lie nearer one line than the other, the space between the lines may be divided into thirds by the eye, and if the button lies in the lower third, add one-third to the percentage corresponding to the lower line, or if in the upper third, subtract one-third from the percentage indicated by the upper line.

The use of the scale has its limits, and it is easy to see that with the increase of weight there is a greater difference for each transverse line, so that there must be a point up to which the determination of the weight on the scale is exact, and beyond which it is better to weigh directly on a delicate balance. The limit depends chiefly upon the amount of practice in placing the button properly with the help of a glass. Experience shows that with ores containing less than 0.5 per cent. the weight of a single button can be more correctly determined on the scale than by the balance, but in a duplicate assay on ores of 0.3 per cent. and upward, the balance gives more accurate results; for ores of over one per cent. the weight of a single button can always be more exactly determined by weighing than by measuring, and when duplicate assays are made the difference is still greater.

The results obtained during practice in measuring the buttons may be controlled by determining the weight of several buttons by measurement, and then actually weighing them all at one time, after thoroughly cleaning them between moist paper and the anvil.

The ivory scale affords a very simple means of measuring buttons, and gives very accurate results in the hands of an experienced operator. By immersing the silver buttons in dilute sulphide of ammonium they become blackened, and then their outlines can be more distinctly seen upon the ivory scale. Unless, however, the operator has become quite expert in its use, by controlling his results with weighed buttons, as before recommended, it is liable to give inaccurate values, and a certain personal error is also apt to occur in the readings, since one person may obtain too high results and another too low ones with the same instruments.

Influenced by a desire to invent an instrument which should yield more certain results than could be obtained by Plattner's scale, Chas. C. Rueger has devised the apparatus shown in Figs. 29 and 30.

This apparatus is described in the *Berg- und Huttenm. Zeitung*, 1869, No. 29, as follows: Fig. 29 shows a horizontal projection of the apparatus; Fig. 30, a section through *C D*. Upon the upper surface of the plate, *a*, is fastened a female screw, *b*, into which the micrometer-screw, *c*, provided with a graduated circle, exactly fits; *e* is a socket, which serves to hold and guide the cylinder of the small slide, *s*. The slide and micrometer-screw touch at *r*. The other wedge-shaped end of the slide rests on the plate at *a*, and has a perfectly plane surface, perpendicular to *a*. Against this presses the likewise plane face of the projection attached to the end of the index lever, *f*; *h* is a spring, which presses the short arm of the lever against the slide. Around the sides of the plate, *a*, is a rim, serving to prevent the button from rolling off. Upon this rim, at *i*, there is a fine line, with which the point of the index lever coincides when the faces of the slide and lever are exactly parallel to each other, *i. e.* touch each other, if nothing is interposed between them.

The graduated circle, *m*, upon the micrometer-screw can be set in any position and secured there by means of the screw, *n*. The periphery of the circle is divided into hundredths, and the index, *o*, should point at 0 on the circle when the lever coincides exactly with the line *i*. The latter condition is secured by using a strong magnifying-glass. When the apparatus has been thus arranged the micrometer-screw is drawn back, whereupon the spring, *v*, presses back the slide, and the spring, *h*, the shorter arm of the lever. The point of the lever at the same time moves away from *i*, until finally the longer arm rests against the pin *w*. If the screw is still further withdrawn the face of the slide recedes more and more from that of the lever, so that a button may be placed between the two. The screw is then turned in the opposite direction, until the point of the lever again coincides exactly with *i*, when the two faces are once more parallel, but separated by an interval corresponding to the distance between their points of contact with the button. This distance is read off in complete revolutions and hundredths of revolutions of the gra-

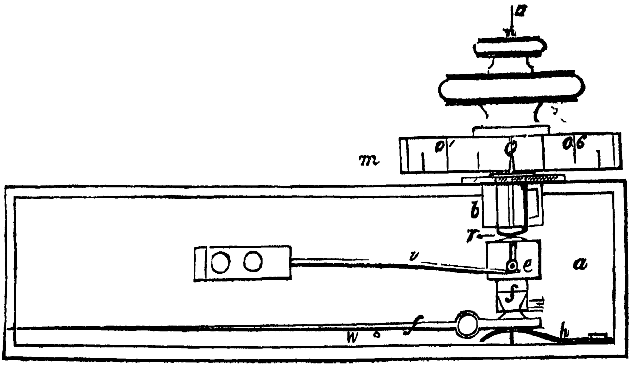


Fig 29

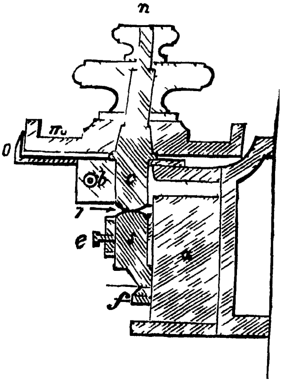


Fig. 30

duated circle; the complete revolutions being indicated by the position of the face of the slide with reference to the lines on *a*, and the hundredths given upon the circle. The interval between two adjacent lines of the scale on *a* corresponds to two revolutions of the screw.

It is advisable to measure the button in several different positions, so as to obtain an average value. To avoid any error arising from possible inequalities of the surface, a fine line is drawn upon the surface, *a*, in the direction of the axis of the screw, and upon this the button should always be placed.

The accuracy of the measurements depends immediately upon the proportion between the longer and shorter arms of the lever. If the line, *i*, and the point of the index lever are fine and sharp enough, and the dimensions of the instrument made to correspond with those in the figure, there will rarely be a difference of one division on the circle, upon repeating the measurement. *In Rueger's instrument the micrometer-screw makes about five revolutions to the millim.*, and therefore advances $\frac{1}{5}$ millim. by one revolution, or $\frac{1}{500}$ millim. for each division of the circle; the results obtained by the inventor seldom varied more than $\frac{1}{1000}$ millim. Such a result is all that can be desired when it is considered that the irregular shape of even an apparently quite round button may lead to five or six times that difference.

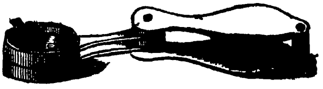


Fig. 31.

4. **A good Magnifying-glass.**—This indispensable instrument is chiefly used to judge more certainly the results of experiments on reactions, and to measure the silver and gold buttons obtained by qualitative assays. A glass well suited for this purpose is composed of two lenses of equal magnifying power, but so mounted that each glass can be used alone, or one brought over the other, so as to use them together. Fig. 31 represents such a double glass.

5. **Forceps and Pliers.**—Various forceps are required for blowpipe assays, viz.,

a. Forceps with platinum tips, for holding an assay directly in the blowpipe flame, when testing its fusibility and other reactions. The forceps, which should be about one hundred and thirty millim. long, are shown in Fig. 32.

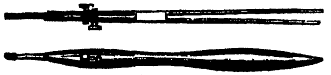


Fig. 32.

b. Cutting Pliers, or *Nippers*, Fig. 33, such as Berzelius employed for breaking off small assay pieces from the minerals to be examined, without injuring the specimens. They resemble nail-nippers, except that the cutting edge is broad and stout, rather than sharp.

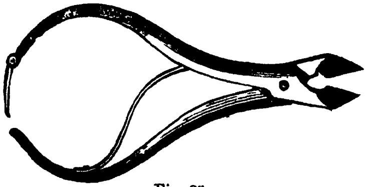


Fig. 33.

c. Steel Pliers, Fig. 34, necessary in separating the slag from the raw lead obtained in gold and silver assays, and for other operations. The jaws of these forceps must be somewhat broad, and the inner surfaces should not be cut like a file, but should only be rough.

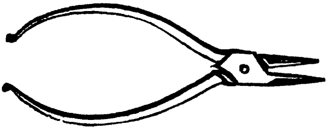


Fig. 34.

d. Brass Forceps, Fig. 35, used for holding small objects, especially in qualitative assays.

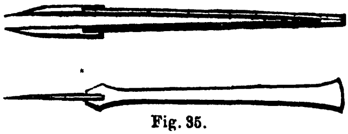


Fig. 35.

e. A similar pair of forceps, also of brass, but somewhat smaller, and having ivory tips; they serve for handling the weights, and also the gold and silver buttons, when measuring them upon the scale.

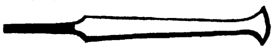


Fig. 36.

f. Iron Forceps, Fig. 36, about one hundred and ten millim. long, which are used in cleaning the lamp-wick and raising or lowering it in the socket.

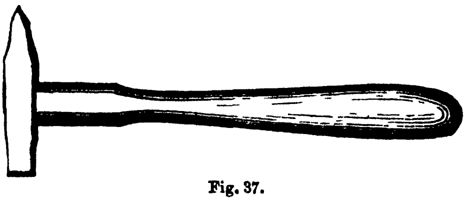


Fig. 37.

6. **A Hammer**, of good hard steel, not too small, rectangular in section, with a polished flat face at one end and a broad edge at the other, as in Fig. 37.

7. **An Anvil**, of hard polished steel, Fig. 38, is used for coarsely breaking minerals and products which are to be pulverized, and also to flatten reduced metallic buttons, to remove the slag from the lead in gold and silver assays, etc. The best form is a parallelopipedon, about fifty-five millim. long, thirty-two millim. wide, and thirteen millim. thick.

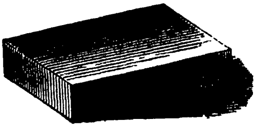


Fig. 38.

8. **A Steel Mortar.**—Abich's mortar, Fig. 39, is best adapted for breaking up and pulverizing refractory metallic minerals, products, and various substances which have been melted on coal before the blowpipe. In the circular plate of hard steel, *A B*, is a cylindrical cavity, *C*, six millim. deep, into which the hollow iron cylinder, *D E*, twenty-one millim. high, with an exterior diameter of twenty-four millim., exactly fits, and into this again fits a stout solid cylinder of hard steel, *F*, forty-five millim. high and eighteen millim. in diameter, which is rounded at its upper end. Both cylinders are turned so as to fit each other exactly. The substance to be pulverized is placed within the cylinder, *D E*, the pestle, *F*, set in upon it and struck a few times with the hammer, while both cylinders are pressed against the steel plate with the fingers. Upon removing the two cylinders, one after another, the substance will be found reduced to a rather fine powder, which may be rubbed still finer in the agate mortar.

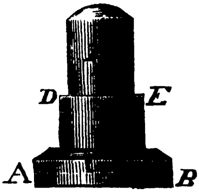


Fig. 39.

9. **An Agate Mortar**, Fig. 40.—By pulverizing very hard bodies in such a mortar it receives, in time, fine scratches, into which some metal is liable to be rubbed, when powdering and washing metalliferous slags, and it must then be cleaned each time with moistened bone-ash.

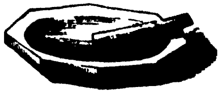


Fig. 40.

10. **A few Files**, triangular, flat, half-round, and round,

varying in size and fineness, are used for different purposes. A rasp is also of excellent service in shaping coals and cleaning them after use.

11. **A Knife** and a small pair of **Scissors,** with strong cutting edges.

12. **A Steel Magnet,** in the shape of a square bar about eighty-five millim. long and four millim. square, wedge-shaped at one end.

13. **Coal-borers.**—In quantitative assays holes must be bored *in the coal, for which various borers are required. Three different ones are used, viz.,*

a. A borer, Fig. 41, to bore holes in the coal for the fusion of silver, gold, copper, and other assays. It is square, and the sides are filed so that it looks like a double chisel, the edges crossing each other at a right angle and being very slightly convex. The breadth of each chisel is eight millim. and the borer is provided with a wooden handle. The borer is placed at a right angle against a cross-section of the coal and turned rapidly on its axis, with a moderate pressure, alternately toward the right and left, until the required depth is reached. After removing the borer the dust is blown out of the hole, the width of which depends on the size of the borer, while its depth is regulated by the height of the paper cylinder in which the assay to be fused is packed. Thus, in case of an assay charged for copper, the hole is shallower than with the charge for an assay of silver ore rich in copper, because the latter contains much test lead.

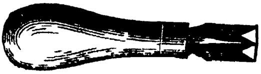


Fig. 41.

b. A conical borer, for boring larger holes, the longitudinal section of which is a semi-ellipse. Its upper diameter is twenty-two millim. and its length eighteen millim. The further arrangement of it is shown in Fig. 42. The instrument is used just as the preceding borer, but as soon as the side, *a,* reaches the level of the perforated side of

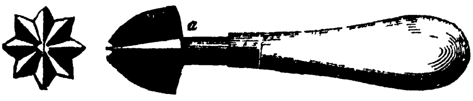


Fig. 42.

the coal the boring is stopped and the coal dust cleared from the hole.

c. A long coal-borer, Fig. 43. One end consists of a double chisel six millim. broad, made like the first borer for cylindrical holes. It is used in boring through the front side of the coals fastened in the coal-holder to be described below, and for boring through the coal covers which are required when fusing quantitative lead, bismuth, tin, nickel, and cobalt assays in clay crucibles. The other end, which is nine millim. wide, is shaped like a spatula, with a sharp edge, and is used for boring holes in coal for qualitative analysis, when they are to be rather deep.

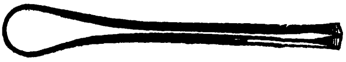


Fig. 43.

14. **Cupel Moulds,** with the **stamps** and **stand.**—For cupelling the argentiferous and auriferous lead obtained from silver and gold assays small cupels of bone-ash are required, which are conveniently made by striking them in a metallic mould and using them for cupellation, without removing them from the mould (Fig. 44). *B* is the stamp, and *A* the mould. The moulds are of iron and are seventeen millim. in diameter, the cavity being worked rough, so that the cupel struck in it may not fall out, as would be very likely to happen if the cavity were smooth. The stamps are of hardened steel, and like the hollows in the moulds their shaping faces are segments of spheres, but of a somewhat larger diameter, and polished. That the mould may be more securely held when hot, a cross is filed on the under side, so that one point of the forceps may be put into one of the four openings, while the other point is pushed over the upper part of the mould, which can thus be transferred to any desired place.

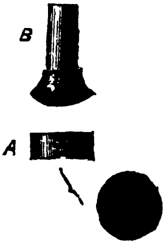


Fig. 44.

The cupels are made by pressing the mould full of bone-ash, setting the proper stamp vertically upon it, and compressing the bone-ash with a few strokes of the hammer, until the convex face of the stamp touches the inner edge of the mould ninety millim. high, Fig. 45, supports the cupel during the cupellation. The cupel mould is then placed on the cross above, so that the cross cuts in the mould do not coincide with the cross pieces, but come between the arms, in order that the hot mould may he removed with the forceps when the cupellation is done.

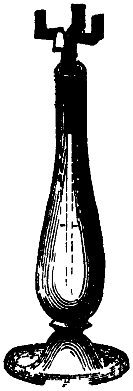


Fig. 45.

15. **A Mixing Capsule** of sheet-brass or horn, polished on the inside and shaped like Fig. 46. It is fifty-eight millim. long, twenty-two millim. wide in the widest part, and five millim. deep, and at the lip, where it begins to round off, seven millim. wide and three millim. deep. It is used, especially in quantitative assays, to pour the mixed charge conveniently into the soda-paper cylinder.

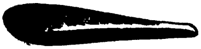


Fig. 46.

16. **A Spatula** of polished iron, of the shape shown in Fig. 47, and ninety-five millim. long. It is used for mixing charges, but more particularly in roasting ores to be quantitatively assayed for metals, and for various other purposes.

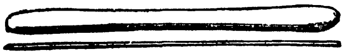


Fig 47.

17. **Coal-holder** with platinum wire and shield.—In making quantitative assays which must be roasted, or fused in clay crucibles, and require a strong heat, the coal used must be protected, at the end employed by an envelope of sheet-iron, the coal-holder. Fig. 48 shows two sides of this holder, as proposed by Plattner. Each of the four sides is thirty-two millim. wide and thirty-six millim. high. On the front side, *B*, is a slit, *b*, ending in a round opening, *a*, seven millim. in diameter, and on the rear side is an iron screw, *c*, on the inner end of which is a plate, *d*, that turns on its axis, while to the outer end the wooden handle, *e*, is attached. The screw, *c*, is

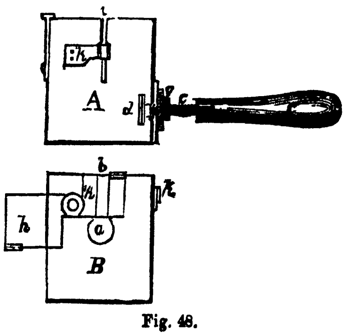


Fig. 48.

below the middle of the coal-holder, so that when the coal is burned out the pressure may not cease, nor the coal fall from the holder. The nut, *f*, outside of the coal-holder, in which the iron screw works, is arranged to slide in and out. There is a small piece of sheet-iron, *h*, fastened on the front of the coal-holder by a small rivet, so that it can be turned to close the slit, *b*, or leave it open, as the dotted lines show. In one side, *A*, of the holder there is also a small slit, *i*, eight millim. long and 0.8 millim. wide, for the insertion of the platinum wire, to be described directly, and below the slit is a small brass socket, *k*, in which the end of this wire is inserted. When an ore is to be roasted in a clay capsule, or an assay fused in a clay crucible, the capsule or crucible must be so placed as to stand free in the cavity and not touch the coal. This is accomplished by means of a platinum wire eighty-three millim. long, and 0.6 to 0.7 millim. thick, which is bent with the pliers into the shape shown in the natural size in Fig. 49. The ring, *A*, is first formed, then, at *l*, the straight part is bent back somewhat, and upward at an obtuse angle, corresponding to the inclination of the sides of the cavity in the coal, and lastly, the remaining part is bent downward at a right angle, as is seen at *B*. The length of the upper horizontal portion must, before bending the wire, be measured off, equal to the distance from the opening in the socket, *k* (Fig. 48), to the side of the cavity in the coal. On the wire, opposite the opening, is hung a small shield of thin platinum foil, Fig. 49, *C* (natural size), which is used *only in roasting*, and serves to prevent the coal from burning out too soon at the part most exposed to the pointed flame.

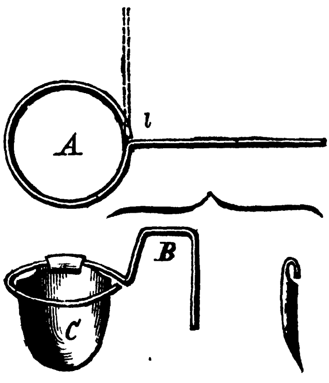


Fig. 49.

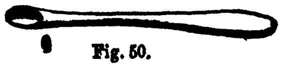


Fig. 50.

18. **A small Ivory Spoon,** eight millim. wide on the exterior and shaped like Fig. 50, quite smooth and polished: also a

small brush for cleaning the scale-pans, mixing capsules, and roasting capsules from any adherent fine dust.

19. **A Test-lead Measure.**—It is troublesome to weigh out the test lead required for a quantitative gold or silver assay, and since *it makes no great difference if a trifle too much or too little is used, Harkort employed a measure similar to that used for gunpowder. It consists of a glass tube,* Fig. 51, thirty-five millim. long and seven to eight millim. wide, ground smooth at both ends, into which a wooden cylinder exactly fits. On the cylinder are several divisions which have been before determined by weighing out five, ten, fifteen, and twenty blowpipe centners of fine test lead and pouring it into the tube. To charge an assay with ten ctrs. of lead the wooden cylinder is drawn out until the line marked 10 is just even with the bottom of the tube, when the empty space above will hold just ten ctrs. of test lead, supposing that the lead used is of the same degree of fineness as that used in determining the divisions.

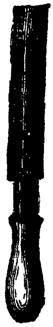


Fig. 51.

20. A small solid **Cylinder of wood**, which is used for preparing the soda-paper cylinders, p. 24. It is twenty-five millim. long and seven millim. thick, as shown in Fig. 52, *B*. To prepare the paper cylinders the wooden cylinder is laid on the paper, Fig. 52, *A*, and the latter rolled around it. The projecting end of the paper is then pressed down on the wood in several places with the little ivory spoon, and finally the closed end is pressed against the table so as to shut it tighter.

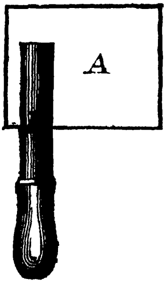


Fig. 52.

21. **Several Cylindrical Boxes of hard wood,** Fig. 53, for dry reagents, and several glass bottles with well-ground stoppers, Fig. 54.

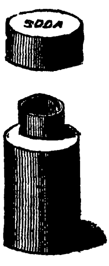


Fig. 53.

Fig. 54.

22. For more extended blowpipe examinations which often necessitate the employment of the wet process, various pieces of apparatus and utensils are required which will be here men

tioned together. *Glazed porcelain vessels,* Figs. 55, 56, 57. Those like Fig. 55 are most suitably made of two sizes: the larger ones about thirty millim. high and forty-five millim. wide in the middle, and the smaller ones twenty-five millim. high and thirty millim. wide. A few watch-glasses of corresponding diameter serve to cover these vessels. Different sizes of Fig. 56 are likewise to be recommended, and paint or indian-ink saucers can be used for the purpose. The smallest size, Fig. 57, twenty-five millim. wide and ten millim. deep, is used to heat, over the spirit-lamp, substances which attack platinum. For dissolving compounds in acids, test tubes, Fig. 58, are used, which are kept in a folding frame of wood or tin. Several small beakers may be advantageously used for similar purposes, as well as for filtrations. A few small glass funnels, which can be placed on the test-tubes, are indispensable; for larger funnels a small filter-stand, Fig. 59, is useful. The filter-paper used must leave but a small amount of white ash, since the ignition of a part of the filter is sometimes unavoidable when trifling precipitates are to be further examined. A small *glass pipette,* Fig. 60, about one hundred and fifty millim. long and blown out in the middle into a bulb,

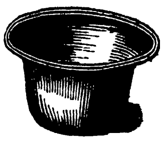


Fig. 55.

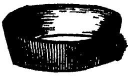


Fig. 56.

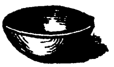


Fig. 57.

Fig. 58.

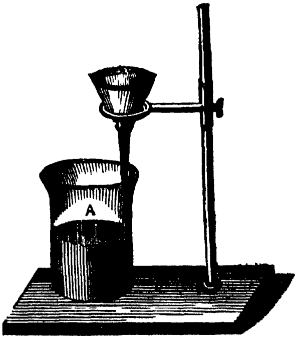


Fig. 59.

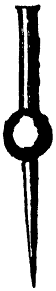


Fig. 60.

twenty-five millim. wide, is also useful. When this is lacking, a tube, ten millim. wide, drawn out to a point at one end, can be used.

Finally, a small *wash bottle*, Fig. 61, or a *dropping glass*, Fig. 62, is necessary. By blowing air into the tube, *a*, Fig. 61, which opens close under the cork, a steady stream of water is forced out through *b*. A glass flask of about forty-five millim. diameter is quite large enough. On inclining the spout of the half-filled dropping glass, Fig. 62, so far that the water runs into it, a few drops will force their way out separately, which is of advantage when anything is to be moistened with a drop of water. It is only necessary to direct the spout downward and blow through the glass tube to force the water out in a very fine stream.

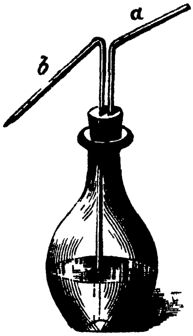


Fig. 61

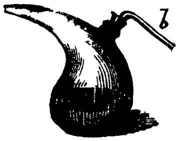


Fig. 62.

23. **Various Tin Vessels,** in case the apparatus is carried during a trip, when it is necessary to take a stock of oil, alcohol, coals, and clay vessels. Tin flasks, covered with dark lacquer, serve for oil and alcohol, the openings being closed like those of the blowpipe lamp. Four-sided cases hold the various coals, which must be tightly laid in, as the artificial coals are easily injured by friction or shocks.

Finally, it is necessary so to pack the objects enumerated in a box, that when they are to be taken on a journey the separate ones may be easily found, while the whole apparatus has a compact appearance.

VI. REAGENTS USED IN QUALITATIVE AND QUANTITATIVE BLOWPIPE ANALYSES.

In blowpipe analyses, as in all chemical examinations, it is essential that the necessary reagents should be used in the enumerated here, but in cases of particular importance, the necessary remarks on their purification, the signs of their purity, and the object of their employment will be made.

A. Reagents for blowpipe analyses made without the aid of the wet process.

a. GENERAL REAGENTS.

1. **Carbonate of Soda.**—Both the carbonate and bicarbonate may be used if chemically pure, and free from sulphuric acid in particular. *Preparation.*—Commercial bicarbonate of soda is pulverized, put into a glass funnel loosely stopped with cotton, and the surface, after being made even, is covered with a circle of double filter-paper, so that the edges, which are bent upward, lie tight against the funnel. It is then washed by pouring on a small quantity of distilled water at a time, until the filtrate, acidulated with nitric acid, is not clouded either with nitrate of silver or chloride of barium. It is then very thoroughly dried, pulverized, and kept in a wooden box; or it may be converted into the simple carbonate by igniting it gently in a shallow porcelain dish, or a porcelain crucible, over a spirit-lamp with a double draught. It must then, however, be kept in a glass-stoppered bottle.

Tests and Use.—A small quantity of the purified salt is treated with the R. F. on charcoal, until it has sunk into the coal. When cold, the spot is cut out, and the mass laid on bright silver foil and moistened with enough water to wet the foil also. If free from sulphuric acid, the surface of the silver remains unaltered, but if a trace is present in the salt, it is reduced with the soda to sulphide of sodium, by the treatment on coal, and causes the silver to become yellow, brown, or black from the sulphide of silver formed, either very soon, or after a few minutes.

Soda serves as a test for sulphuric acid, and also, in qualitative and quantitative analyses, to decompose compounds of silicic, tungstic, and titanic acids, and to promote the reduction of various metallic oxides.

2. **Neutral Oxalate of Potassa.**—Binoxalate of potassa is dissolved in water, neutralized with potassa, filtered, and evaporated to dryness, being stirred with a glass rod toward the

end of the operation, while a sufficiently high temperature is employed, but not enough to decompose the salt. The dry mass is pulverized and kept for use in a well-closed bottle.

Tests and Use.—The solution of the neutral salt must remain unclouded on adding sulphide of ammonium. When completely dry, this salt, at a low red heat, evolves carbonic oxide, without carbonic acid, and is, therefore, admirably suited to the examination for arsenic, and is better than soda for reduction assays on coal.

3. **Cyanide of Potassium.**—Liebig's method is to rub eight parts by weight of anhydrous ferrocyanide of potassium, free from sulphate of potassa, with three parts of dry carbonate of potassa, and fuse the mixture at a moderate red heat in a covered porcelain crucible until the mass is clear and a sample taken out appears quite white. The clear mass is carefully decanted from the fine separated iron at the bottom upon a bright piece of sheet-iron, and the solidified salt is kept in the form of coarse powder in a glass-stoppered bottle.

Use.—The salt prepared as above contains some cyanate of potash, but this does no harm. It is chiefly used as a reducing agent, being in this respect always preferable to soda, and in many cases to oxalate of potash.

4. **Borax (biborate of soda).**—Commercial refined borax is perfectly suited to blowpipe assays. The pulverized hydrated salt serves for qualitative analyses, but for quantitative assays, where all intumescence must be avoided as far as possible, it is better to employ the borax in the state of glass. It is fused in a platinum crucible over a spirit-lamp with a double draught, or on coal before the blowpipe, in the O. F., and the glass broken to coarse powder between paper, or in the steel mortar, is rubbed somewhat finer in the agate mortar and kept in a glass-stoppered bottle. Impure borax can be purified by recrystallization and washing the crystals in a glass funnel with a little cold distilled water, after which they are dried and pulverized.

Tests and Use.—A small quantity of pure borax melted in the loop of a platinum wire must not give a coloured bead in the O. F. or the R. F., and dissolved in water it should give no precipitate with carbonate of soda, and after adding nitric acid to its solution there must be no precipitate with nitrate of baryta or nitrate of silver.

Boracic acid has the property of combining with oxides at a high temperature, driving out weak acids, and with the aid of the O. F. disposing metals, and sulphur, and haloid compounds to oxidize. Borates of the oxides result, which fuse readily with borate of soda and give a clear glass. The borax contains, besides borate of soda, free boracic acid, and is therefore inclined to dissolve bases and acids, and to form basic as well as acid double salts, which are all to a certain degree fluid. These salts generally remain clear on cooling, and thus the colour produced by the combination with the dissolved body is the more clearly brought out in qualitative examinations. In quantitative assays it serves, partly alone and partly in common with soda, to dissolve the earths and metallic oxides of difficult reduction occurring in the ores and minerals, as well as to separate various metallic arsenides from one another.

5. **Salt of Phosphorus, or Microcosmic Salt** (phosphate of soda and ammonia).—According to Berzelius, this salt, which is not always obtained free from chloride of sodium, is best made as follows: One hundred parts of crystallized phosphate of soda and sixteen parts of chloride of ammonium are dissolved, with the aid of heat, in thirty-two parts of water, and the boiling-hot solution filtered and allowed to cool. The double salt crystallizes while cooling, leaving in the solution chloride of sodium and some of the double salt, which, however, cannot be crystallized out free from chloride of sodium by further evaporating the mother liquor, and cannot, therefore, be used for blowpipe examinations. After completely decanting the mother liquor, the crystals are dried between filter-paper and the salt kept for use in wooden boxes. When heated on coal or platinum wire it boils, swells a little, and gives up its water and ammonia, leaving behind acid phosphate of soda, which melts quietly and solidifies on cooling to a clear colourless glass.

Tests and Use.—If the salt is not free from chloride of sodium it sometimes gives a glass which is not clear on cooling, and when a little oxide of copper is dissolved in the salt which is fused on platinum wire, the outer flame is coloured blue. In this case it must be redissolved in a little boiling water and set aside to recrystallize. When freed from its water and ammonia by fusion, the salt acts particularly through its free phosphoric acid, which exerts a powerful solvent action on many substances under examination. Taking up all bases it forms with them more or less fusible double salts, the transparency and colour of which are chiefly to be regarded. It serves also to separate the bases in the test for fluorine.

While this salt is analogous in its action to biborate of soda, the glasses which it forms with metallic oxides generally differ in colour and intensity from those obtained with borax and the same oxides by similar treatment.

6. **Nitre (nitrate of potassa).**—Commercial saltpetre is dissolved in a small quantity of boiling water, and the boiling-hot solution filtered and set aside to crystallize. After a small quantity of crystals has formed, the mother liquor is poured off and the crystals dried between filter-paper and kept in wooden boxes.

Nitre serves only as an oxidizing agent.

7. **Bisulphate of Potassa.**—This salt can be prepared thus: One part by weight of pure sulphuric acid is poured over two parts of coarsely-broken crystals of pure sulphate of potassa in a porcelain crucible, which is then gradually heated over a spirit-lamp until the whole forms a fluid clear as water, when the crucible is removed and the fluid salt allowed to cool. It solidifies very quickly, appears quite white, and can be obtained in one piece by turning the crucible over when cold. The pulverized salt is preserved for use in a glass-stoppered bottle.

Use.—It is used in testing lithium, boracic acid, nitric acid, fluorine, bromine, chlorine, and iodine, and for decomposing compounds of titanic, tantalic, and tungstic acids. It can also be used to separate baryta and strontia from the other earths, and various metallic oxides by the wet way, when the blowpipe alone is not sufficient. The method of using it will be especially described under the separate assays.

8. **Vitrified Boracic Acid.**—This can be obtained from the chemical works of as good quality as it can be prepared, and more cheaply. It is kept in the form of powder, or in small fragments, in a tightly-closed bottle.

It serves in qualitative examination to detect phosphoric acid in minerals, and a small quantity of copper in presence of much lead. In quantitative assays it is quite indispensable in separating lead from copper, and from alloys of the noble metals, which fuse with difficulty and cannot be quite freed from it on the cupel.

9. **Nitrate of Cobalt in solution.**—Pure protoxide of cobalt is dissolved in the necessary quantity of dilute nitric acid, the solution evaporated to dryness at a gentle heat, the dry salt dissolved in water, and the filtered solution kept in a bottle. The solution should not be concentrated, as this would not answer the purpose in all cases. The protoxide of cobalt must be chemically pure, containing neither protoxide of nickel nor sesquioxide of iron, and there must be no potassa adhering to it. The latter can be removed by boiling the protoxide with distilled water.

Use.—The nitrate of cobalt serves for recognizing various earths and metallic oxides with which protoxide of cobalt forms, on being heated to redness in the O. F., compounds distinguishable by their peculiar colours.

As only one or two drops are required for an assay, it is convenient to take out the requisite amount with a small instrument, and thus moisten the substance to be examined. This is most simply effected with a platinum wire, spoon-shaped at one end, and fixed in a cork, or with a thin glass tube, likewise fastened in a cork which fits the neck of a small bottle. The latter is very convenient, since the tube acts like a pipette when it is dipped into the solution and the cork pressed a little into the neck of the bottle, the compression of the air forcing some of the solution into the tube. On drawing out the cork and the filled tube, and closing the wide end of the latter with the finger, one, two, or several drops may be allowed to escape without touching the substance with the narrow end of the tube.

10. **Test Lead**, finely granulated, and also in lumps, as free as possible from other metals, particularly gold and silver.—When the granulated test lead can be obtained from lead and silver works it need only be sifted through a small sieve, and the part which passes through is kept in a wooden box. If

such lead cannot be had, the requisite amount may be prepared by reducing the lead from acetate or sugar of lead with zinc, in the following way: Dissolve sugar of lead in a small quantity of boiling water, then place a zinc rod in the filtered solution, and after about six hours, carefully detach the metallic lead from the rod, so as to expose a fresh surface, repeating the operation every six hours, until all the lead is separated in the metallic state. The lead is then purified from the adherent acid solution, which contains zinc, by repeated washing, and dried between filter-paper in a warm place, after which it is rubbed in a porcelain mortar and the fine part separated from the coherent mass with a sieve.

Test lead is chiefly used in quantitative gold, silver, and copper assays. The volume of a weighed amount of test lead prepared from sugar of lead is to the volume of an equal weight of granulated and sifted lead as six to five, and where the former lead is to be used by measurement for silver and gold assays with the measure, and not by weight, there must be added one ctr. for every five ctrs. in order to obtain the proper weight. When the granulated lead cannot be easily obtained, the preceding method with sugar of lead affords the readiest means of preparing pure and finely divided test lead.

11. **Tin.**—Ordinary tinfoil is used, cut into long strips twelve millim. wide and rolled up tight.

Tin serves to produce the lowest degree of oxidation in glass fluxes, particularly in case of trifling quantities of such metallic oxides as can be reduced to protoxide or suboxide, and in this state give more conclusive results. A little of the tin is cut off, laid directly against the glass bead and quickly fused, for a moment only, in the R. F. After adding the tin the blast must not be kept up too long, partly because the tin, will entirely reduce many metals which should only be brought to the lowest state of oxidation, and thus recognized by the colour of the glass, and partly because so much tin may be dissolved, especially with S. Ph. that the glass becomes quite opaque, whereupon the reaction disappears entirely.

12. **Iron,** as fine piano wire, and also wire as thick as a moderately coarse knitting-needle.

The fine wire serves in the test for phosphoric acid, while the stout wire is used in the qualitative test for antimony and in the quantitative lead assay.

13. **Silver.**

A small piece of sheet silver is used in reactions for sulphide of sodium or soluble sulphides. It is also required in many quantitative gold assays and can be best reduced from chloride of silver, being hammered and rolled into a thin sheet from which the desired amount may be easily cut.

14. **Gold,** in small buttons, up to eighty milligr. in weight, or as foil.—It may be had pure by dissolving gold coin in nitro-muriatic acid, diluting the solution properly with water and letting it stand until it is clear and the small amount of chloride of silver has settled, when it is filtered, and the gold precipitated with sulphate of iron. After the finely-divided gold has settled, it is collected on a filter, well washed, dried, and gently ignited. It is best to ignite the filter separately. From this gold, buttons of any desired size can be made by fusion on coal with a little borax.

Gold serves in many reduction assays to collect small quantities of reduced metals, which are to be simply separated, or recognized, or else, as is frequently the case, are to be quantitatively determined; viz. copper and nickel.

15. **Arsenic** in the metallic state.—If it cannot be purchased, powdered mispickel is treated in a glass retort provided with a receiver, as long as a sublimate of metallic arsenic collects on the neck of the retort. When cold, the retort is broken and the arsenic which has collected toward the end and is free from sulphur is kept for use.

It serves in the nickel and cobalt assays to convert the metallic oxides into arsenides.

b. SPECIAL REAGENTS USED ONLY IN CERTAIN EXAMINATIONS.

1. **Test-papers** of blue and red litmus and Brazil-wood paper cut into small strips.—The paper is coloured in the following way:

a. Blue litmus-paper.—One part of litmus, of the best quality, is rubbed to a coarse powder and mixed with water to a paste, which is tied up in a bag of fine linen and hung in a vessel containing ten times as much boiling water as the weight of the litmus employed. The colouring matter is extracted, forming a solution which is poured into a porcelain dish, and the strips of paper, after being drawn through it with a glass rod, are hung upon a cord to dry, in a shaded place free from dust. Fine filter-paper in strips two inches wide is best suited for this purpose.

b. Red litmus-paper.—A litmus solution prepared as above is reddened with the least possible amount of dilute sulphuric acid, added very gradually while the liquid is strongly stirred, so that no more acid is added than is exactly necessary, as otherwise the paper is not sensitive enough. The paper is then prepared as before.

c. Brazil-wood paper.—Shavings of Brazil-wood are boiled in a glass flask with water, the liquid decanted from the shavings and paper coloured with it in the same way as with the litmus extract.

Use.—Blue litmus-paper is used for recognizing free acids; red litmus-paper is a sensitive reagent for free alkalies. Brazil-wood paper is used especially in the test for fluohydric acid, which imparts to it a yellow colour.

2. **Antimonate of Potassa**, in a pulverized state.—One part of antimony is mixed with six parts of nitre and deflagrated in a red-hot clay crucible. The resulting mass is pulverized, lixiviated with cold water, and then treated with water at a boiling heat, which dissolves the neutral antimonate of potassa, and an acid salt remains. The solution of the neutral salt is separated from the residue by filtration, evaporated to dryness, and the dry salt heated strongly, until it is quite free from water and has assumed a white colour. It is pulverized while warm and preserved in a well-closed bottle.

It is employed to detect small quantities of carbon in compound bodies. When ignited with them it yields oxygen to the carbon, forming carbonate of potassa, which gives up its carbonic acid in the form of gas when dissolved in water, and decomposed by nitric acid while still quite warm.

3. **Common Salt**, or, **Rock Salt** (chloride of sodium); decrepitated, or fused and powdered.

Its use is very limited, but it is employed with advantage as a cover for the necessary charges in quantitative lead, bismuth, tin, nickel, and cobalt assays, when they are fused in clay crucibles.

4. **Fluor Spar,** heated to redness and kept in a wooden box. —It must be quite free from boracic acid, which, according to Kersten, is not always the case. When mixed, therefore, with bisulphate of potassa and fused on platinum wire, in the blue flame, it must not colour the outer flame green.

It serves, in connection with bisulphate of potassa, to detect lithium and boracic acid in compounds.

5. **Quartz (silicic acid).**—Pure silicic acid, separated from the silicates in the course of chemical analysis, may be used, provided it has a perfectly white colour after ignition. Rock crystal may also be used, being coarsely powdered between paper, and then pulverized in an agate mortar.

Fused to a glass with soda it serves to test substances for sulphuric acid, and, in combination with soda and borax, to separate tin from copper; also frequently, in connection with soda, to test for phosphoric acid.

6. **Oxide of Copper.**—It is most simply prepared by dissolving pure copper in nitric acid, evaporating to dryness, and gradually heating the dry mass to a strong red heat in a thin porcelain dish.

It serves chiefly to detect small quantities of chlorine in compounds.

7. **Oxalate of Nickel.**—Protoxide of nickel, quite free from cobalt and iron, is dissolved in hydrochloric acid, the solution evaporated to dryness on a water-bath, the dry mass dissolved in water, filtered, and the oxide of nickel precipitated with oxalic acid. The precipitate is dried and kept for use. It serves to detect potassa in certain cases.

8. **Chloride of Silver,** in the form of a thick paste.

It serves to produce certain coloured flames, which appear more distinct with it than with hydrochloric acid.

9. **Starch Meal.**

It acts as a reducing agent in quantitative lead, bismuth, tin, nickel, and cobalt assays, when they are fused with carbonate of potassa and soda in clay crucibles.

10. **Graphite.**—Commercial graphite is often very impure, usually containing many earthy particles, and cannot then be used for blowpipe assays. A piece must be sought which is soft, but not scaly and lamellar, for such graphite burns with difficulty. It is pulverized and its purity tested by burning a little of the powder in a clay capsule. When a pure piece cannot be obtained, the impure graphite is purified in the following way: After being pulverized as fine as possible, it is mixed with twice its volume of carbonate of potassa and heated to redness in a covered clay crucible. The heated mass is then powdered and boiled with water, and any admixture of quartz thus removed with the alkali: by heating the remaining graphite with dilute nitric acid the iron and earths can be dissolved and separated by filtration and thorough washing. The graphite which remains is thoroughly dried and kept for use.

Pure, very finely-divided graphite serves well as an addition *in roasting substances containing sulphur or arsenic in which the copper is to be quantitatively determined.*

11. **A Touch-stone** *for testing the precious metals and their alloys.*—It consists of hard black basalt or flinty jasper ground smooth. The metallic streaks are removed by smearing the stone with a little oil and rubbing it with charcoal, after which the surface is wiped off, and appears clean and quite black again.

B. Reagents for blowpipe analyses which are performed with the aid of the wet process.

a. GENERAL REAGENTS.

1. **Sulphuric Acid,** concentrated.

It serves to intensify the reactions of boracic and phosphoric acids in the outer flame, with substances containing those acids; to decompose many compounds of fluorine, and in the examination for arsenic. It is used in a dilute state to detect lime, under certain circumstances, and also to separate baryta and strontia from other earths.

2. **Nitric Acid,** chemically pure.

It is used to peroxidize protoxide of iron in solutions, to separate silver from gold, and is of advantage in other cases.

3. **Hydrochloric Acid.**

It serves to detect small quantities of ammonia in salts; the ammonia being set free by soda, under the influence of heat, and brought into contact with a glass rod moistened with the acid, when a white cloud is produced. It also serves to detect carbonic acid in compounds soluble in hydrochloric acid. Further, to dissolve various earthy salts, to decompose many silicates, the bases of which cannot be certainly determined by the blowpipe alone; and finally, in the quantitative tin assay, to separate various metallic oxides from the roasted tin ore.

4. **Acetic Acid.**

Tests and Use.—Acetate of baryta must not cause any cloudiness. It is only used in examining compound substances for chromic, vanadic, and phosphoric acids.

5. **Oxalic Acid.**

When dissolved it serves to precipitate lime from an ammoniacal liquid, and to separate zirconia, sesquioxide of iron, and sesquioxide of uranium from yttria and the oxides of cerium and lanthanum. It can also be used to reduce gold dissolved in aqua regia.

6. **Caustic Potassa,** in solution.—It can be carried in the solid state during a trip and dissolved in the necessary amount of water when used. The commercial potash, not always quite free from alumina, may be purified by dissolving it in absolute alcohol, decanting the solution from the residue, and concentrating it in a silver vessel until it forms a glowing fluid mass, which is then poured on cold, clean sheet-iron, and after solidifying, preserved in a glass-stoppered bottle.

Dissolved in water it serves to separate alumina and glucina from the sesquioxides of iron, manganese, and chromium, after they have all been precipitated from their solutions by ammonia and thoroughly washed.

7. **Ammonia.**

Tests and Use.—It must be free from carbonic acid, so that a solution of chloride of calcium must produce no cloudiness, by the formation of carbonate of lime. It is used in blowpipe analyses, with the aid of the wet process, when the separate earths are to be detected in compounds, for the purpose of separating alumina, glucina, yttria, and the sesquioxide of chromium, iron, etc., from lime, magnesia, and protoxide of manganese in solutions, which in many cases must contain free hydrochloric acid, or hydrochloric acid, or chloride of ammonium.

8. **Carbonate of Ammonia.**—It is kept in a powdered state in a glass-stoppered bottle.

Dissolved in water it serves, in analyses which cannot be performed with certainty before the blowpipe alone, to separate glucina from alumina, and sesquioxide of uranium from sesquioxide of iron, in the wet way. It is also added to the water used in washing precipitated phosphate of ammonia and magnesia, and is employed in roasting many quantitative assays, to secure the decomposition of certain metallic sulphates.

9. **Bichloride of Platinum.**—It is prepared by warming very thin bits of platinum with twice their weight of hydrochloric acid, and adding nitric acid by drops until all is dissolved. To remove the free acid the liquid is carefully evaporated to dryness, the brown residue dissolved in water, filtered, and kept in this state. It must not be too dilute.

It is the best reagent in the wet way for potassa, when occurring in trifling quantities in compounds with soda or lithia.

10. **Distilled Water.**—When the aid of the wet way is required, distilled water must always be at hand. Evaporated on platinum foil it must leave no residue, and must not be rendered cloudy by salts of barium or silver.

b. SPECIAL REAGENTS OF LIMITED USE.

1. **Tartaric Acid,** in a crystallized state.—It is kept in a wooden box, and is used in the separation of iron from yttria and zirconia by sulphide of ammonium.

2. **Carbonate of Potassa.**—It is kept in the dry state in a glass-stoppered bottle.

It replaces soda in the qualitative test for tantalic and niobic acids, and may also be used in connection with soda for quantitative assays that must be fused in clay crucibles.

3. **Neutral Sulphate of Potassa,** in small crystalline crusts.—It is obtained quite pure from the apothecaries, and kept in a wooden box.

It is frequently employed to detect zirconia, and to separate the oxides of ceriúm, lanthanum, and didymium from other bodies by the wet way, when the blowpipe alone is not sufficient.

4. **Chloride of Ammonium** (**sal ammoniac**).—It is obtained quite pure from the apothecaries, but may be redissolved in hot water, filtered, and set aside to recrystallize. After decanting the mother liquor the crystals are dried between filter-paper and kept in a wooden box.

It is used only in the assay for gold when platinum or iridium is to be separated.

5. **Molybdate of Ammonia.**—It may be prepared by pulverizing molybdenite as finely as possible, and roasting it in a small and quite shallow dish of platinum or sheet-iron, at a moderate heat, over a gas-lamp, or spirit-lamp with a double draught, until the powder becomes yellow, and is mostly converted into molybdic acid, which turns yellowish-white on cooling. It is then digested for some time with ammonia, which extracts the molybdic acid, forming molybdate of ammonia, and this is separated from the residue of undecomposed molybdenite and binoxide of molybdenum, evaporated to dryness, and kept for use. The residue may be again roasted and treated with ammonia.

Molybdate of ammonia dissolved in water is the most delicate reagent for phosphoric acid.

6. **Ferrocyanide of Potassium.**—This may be kept in the crystallized state, and the requisite quantity dissolved in eight or ten parts of water when used.

It serves to detect a very trifling amount of iron in substances containing chromium and vanadium, when they have been previously fused with bisulphate of potassa.

7. **Sulphide of Ammonium.**—Pure caustic ammonia is diluted with an equal volume of water, and hydrosulphuric acid gas conducted into it until a solution of sulphate of magnesia mixed with a portion of it remains perfectly clear. This test is necessary to ascertain whether the ammonia is quite saturated.

The sulphide of ammonium thus made is kept in a glass-stoppered bottle, over which is fastened a piece of bladder or thin caoutchouc.

It is used to separate protoxide of manganese and cobalt from magnesia, sesquioxide of iron from yttria, and tungstic acid from tantalic and niobic acids, as will be described in the various wet analyses.

8. **Sulphate of Protoxide of Iron.**—To preserve this salt without the formation of sulphate of the sesquioxide, the purest protosulphate of iron is dissolved, according to Otto, in boiling water, the solution boiled with metallic iron, filtered while quite hot, and alcohol added, stirring the solution meanwhile. On cooling, a salt, perfectly free from sesquioxide, separates in small crystals, which are collected on a filter and washed with alcohol, the funnel being covered over after each fresh portion is poured on. After first freeing the purified salt from most of the adherent fluid between filter-paper, it is wrapped in the same kind of paper, dried in a moderately warm place, and preserved in a bottle.

Dissolved in water it serves in many cases to precipitate gold from its solution in aqua regia, since it has a reducing action. There are other agents for precipitating gold, such as oxalic acid, terchloride of antimony, etc., but sulphate of iron is quite sufficient for blowpipe assays.

9. **Sulphate of Copper.**

It is of very limited use, but is frequently employed with advantage to detect chlorine in compounds. It is kept in the shape of coarse powder in a wooden box.

10. **Acetate of Lead**, kept in the crystalline state in a wooden box.

It is used in testing for chromium, vanadium, and phosphoric acid, when the wet way is partially necessary.

11. **Infusion of Galls.**—One part of coarsely-powdered galls is digested for twenty-four hours with six parts of alcohol, the liquid decanted, and the residue pressed out; both solutions are then mixed, filtered, and the clear fluid kept in a well-closed bottle.

It serves to distinguish tantalic from niobic acid, as will be given under the examination for tantalum and niobium.

12. **Absolute Alcohol.**

It is used with bichloride of platinum in testing silicates for potassa, and is also required in the examination for baryta and strontia, both to distinguish these earths and to separate them from one another.

13. **Chloride of Mercury** (corrosive sublimate).

It is only used in the quantitative chromium assay.

GENERAL RULES.

A. General rules according to which the behaviour of *minerals and other substances before the blowpipe can be determined, and a considerable proportion of their constituents discovered.*

THE behaviour of a substance before the blowpipe is tested partly without and partly with reagents. The testing without reagents is performed :—

1. In a small glass matrass, or a closed tube, to learn whether the substance decrepitates when heated, or yields volatile constituents which may be recognized; thus frequently giving indications of the composition of the substance.

2. In an open tube, to detect constituents which oxidize and volatilize on ignition with access of air.

3. On coal, to observe what alterations the substance undergoes in the O. F. and R. F., and whether metallic constituents become volatile, oxidizing and forming a coat on the coal, by which they may be recognized, etc.

4. Either in the platinum forceps, or, if the substance is an easily fusible salt, in the loop of a platinum wire, partly to learn the fusibility of the substance, and partly to learn whether it colours the outer flame, and how.

The testing with reagents is performed partly on platinum wire and partly on coal. As regards the amount or size of the substance to be tested, it can only be generally remarked that for testing in matrasses and tubes, too little should not be used, while in testing the fusibility and colour of the flame only very small splinters or pieces should be taken; and the same is true in testing substances containing metallic oxides with borax and salt of phosphorus.

It is advisable to place the blowpipe-lamp on a sheet of white paper on the table, both to protect the latter and to keep the substances which may fall from being rendered impure, while they can also be more easily found again.

a. Examination of the Substance without Reagents.

I. TESTING IN A SMALL MATRASS, OR IN A CLOSED GLASS TUBE.

When the substance seems free from inorganic combustible bodies, like sulphur, arsenic, etc., a suitable quantity of it is put into a matrass, Fig. 21, A; if such bodies are present, however, then a closed tube, Fig. 21, B, is employed, which must not be too wide, so that the substance is surrounded with as little air as possible, and no oxidation of the combustible bodies may ensue. In both cases the assay is heated in the flame of the spirit-lamp, at first gently, and then to redness if necessary, while all the phenomena are noted. The most usual phenomena occurring in such a test are illustrated by examples in the following pages.

Examination of the substance by itself in a small matrass.

Here belong salts and similar compounds, silicates and aluminates, hydrates, and metallic oxides with their compounds.

α. The substance when heated is altered in shape or condition; decrepitates (*e.g.*, *barite, fluorite, calcite, and many other minerals*); phosphoresces (*fluorite*, especially the green variety, *apatite*, certain *calcites, harmotome, cyanite*); appears to glow as if on fire (*glassy gadolinite, orthite, samarskite, etc.*); or changes its colour, which reappears in many cases on cooling, while nothing seems to volatilize except, perhaps, a little water (*zincite, titanic acid, cerussite*, which turns yellow, *malachite, siderite, oxide of zinc*, which becomes transiently yellow).

β. The S. does not appear to alter, but, after longer and more intense ignition, yields volatile constituents. Thus, many peroxides evolve oxygen, which may be easily recognized by placing a splinter of charcoal on the assay, when the coal suddenly enters into active ignition on being sufficiently heated. *Hydrates, hydrous silicates, or substances which contain water mechanically combined,* yield water, which collects in the neck of the matrass. In case they also contain sulphuric acid or fluorine and are decomposed by heat, the water has an acid reaction on litmus or Brazil-wood paper, after strong ignition; in the presence of a decomposable metallic fluoride, hydrofluoric acid is also formed, which attacks the glass at a short distance from the assay and renders it dull.

γ. The S. fuses without yielding water, and boils when strongly heated.—Certain *chlorates, bromates,* and *iodates.*—The oxygen evolved reignites a glowing splinter introduced into the matrass, or causes a deflagration when coal dust is thrown on the melting salt. *Nitrate of potassa* and *soda* behave similarly.

δ. The S. fuses, yields much water, which collects in the neck, and becomes solid again.—*Salts containing much water of crystallization.*—After drying the neck with paper, a strip of litmus-paper is introduced, and the S. more strongly heated. Many salts with weak bases, as alumina, sesquioxide of iron, &c., are decomposed at a red heat, yielding a more or less acid reaction, and sometimes clouding the glass above the assay. The liberated acids may also sometimes be recognized by their odour.

ε. The S. fuses and yields acid.—*Acid salts, the acids of which are volatile when in a pure or hydrated state.*—The escaping excess of acid reddens litmus-paper. Among the neutral salts with volatile acids only, the nitrates and hyposulphates are generally decomposed; the former yield yellowish-red nitrous acid gas, and the latter colourless sulphurous acid. In certain cases metallic fluorides yield more or less hydrofluoric acid, especially when the compound contains water, and part of the fluorine is combined with a weak base like aluminium. Should violet vapours, with an odour of iodine, appear, they indicate free iodine.

ζ. Charring takes place, sometimes accompanied by intumescence, and often with a burnt or bituminous odour.—*Organic substances.*—If the cold, charred residue effervesces with acids, this indicates a combination of alkalies or alkaline earths with the organic acids in the substance. Certain organic acids can be at the same time detected by the odour evolved while charring; as tartaric acid, benzoic acid, &c. The presence of cyanogen compounds is shown by the odour of hydrocyanic acid evolved on adding hydrochloric acid. Sometimes on heating nitrogenous substances ammonia is formed, which can be detected by red litmus-paper or a rod dipped in hydrochloric acid.

η. The substance sublimes and condenses in the neck.—Most *ammoniacal salts,* which either sublime entirely, or, if combined with a non-volatile acid, simply yield ammonia; *protochloride of mercury,* which first fuses and then sublimes; *subchloride of mercury,* which sublimes without fusing, becoming yellowish, but on cooling is white again; *chloride of lead,* which fuses to a dark, yellow fluid, sublimes partially, and becomes opaque and white on cooling; *oxide of antimony,* which first fuses to a yellow fluid and then sublimes in lustrous needles, unless more highly oxidized by the enclosed air; *arsenous acid,* which sublimes very easily and condenses in a crystalline state; *tellurous acid,* resembling oxide of antimony, but volatilizing with far more difficulty, and forming no crystalline sublimate; *osmic acid,* subliming in white drops, and evolving a penetrating, extremely disagreeable odour.

Examination of the substance alone in the closed tube.

In this way are tested sulphides, selenides, arsenides, tellurides, and metallic compounds.

α. The S. is unaltered, or decrepitates, or fuses and gives nothing volatile, even when heated with the blowpipe until the glass begins to melt. Sometimes the glass becomes clouded just above the assay, but this results from a little sulphurous acid acting on the glass.

β. The S. yields a sublimate of sulphur, dark yellow to reddish-brown while warm, pure sulphur-yellow when cold.—*Metallic sulphides* containing much sulphur, as *iron pyrites,* which yields nearly one atom of sulphur; or combinations of metallic sulphides where one of them has more than one atom of sulphur for one atom of the metal, as *chalcopyrite*. Sometimes the native protosulphides contain a slight excess of sulphur and yield it as a thin, almost white film.

γ. The S. yields a sublimate of sulphide of arsenic, dark brownish-red to almost black while hot, and reddish-yellow to red when cold.—*Native* or *artificial sulphide of arsenic*, and combinations or mixtures of *metallic sulphides* and *arsenides*, which, on giving up all their arsenic contain more sulphur than is required to convert them into protosulphides.

δ. The S. heated strongly yields a black sublimate, condensing just above the assay, and becoming cherry-red to brownish-red on cooling. It is a combination of sulphide and oxide of antimony.—*Sulphide of antimony* and *compounds of metallic sulphides with much sulphide of antimony*.

ε. The S. yields a black, lustrous, metallic, and crystalline sublimate of arsenic, and evolves an alliaceous odour.—*Metallic arsenic* and arsenides containing more than one atom (double atom) of arsenic for two of the metal, as *smaltite, chloanthite*, &c.; also compound sulphides and arsenides in which the liberated arsenic is replaced by the sulphur, which combines with the bases to form protosulphides, as *pyrite*. A little sulphide of arsenic sometimes sublimes first.

ζ. The S. gives a dull black sublimate of sulphide of mercury, which becomes red when rubbed.—Native or artificial sulphides of mercury, as *cinnabar;* also *mercuriferous tetrahedrite*.

η. The S. yields a lustrous, crystalline, gray sublimate of selenide of mercury.—*Tiemannite* and *lehrbachite*.

θ. The S. yields a film or coat of small drops of lustrous mercury.—*Amalgams*.

Non-volatile *metallic sulphides* and such as contain a low proportion of sulphur; non-volatile *arsenides* and those containing only one atom (double-atom) or less of arsenic for two atoms of the metal; further, *tellurides* and *antimonides* yield uncertain results, or none at all, in the closed tube.

2. EXAMINATION OF THE SUBSTANCE IN THE OPEN TUBE.

The substance tested in the closed tube must also be tested in the open tube. The assay is laid near one end of the tube, and the spot heated with the spirit-lamp at first, and then, if necessary, with the blowpipe, the tube being somewhat inclined. Generally fragments are used, but substances which decrepitate are first pulverized. The force of the current of air can be regulated by the inclination of the tube. *Many bodies not volatile in the closed tube here* absorb oxygen from the current of air and form volatile acids or metallic oxides. Some escape as gases, recognizable by their odour; others condense as sublimates in the cool part of the tube, at distances from the assay varying according to their volatility. In this roasting test, too large a piece should not be taken, nor the heat allowed to work strongly upon it too soon, otherwise the greater part of the volatile bodies may sublime unchanged. Should a fragment yield no distinct reaction, the substance must be powdered.

There are several bodies which can be recognized with the open tube, even in very compound substances. The principal ones are the following:—

a. **Sulphur.**—On treating metallic sulphides, or substances containing such sulphides even in trifling quantity, sulphurous acid is found which can be recognized by its odour, or by reddening blue litmus-paper at the top of the tube. Sulphides which roast with difficulty, as blende and molybdenite, with substances containing little sulphur, must be powdered. When the substance contains metals that form volatile oxides peculiar sublimates are formed, the characteristics of which will be described more nearly below.

With too large a fragment or too rapid heating sulphur, sulphide of arsenic, or sulphide of mercury, may sublime from the corresponding substances tested, because air enough is not present to oxidize them.

β. **Selenium.**—Selenides and substances containing even trifling quantities of selenium yield a gaseous oxide with the odour of decaying horseradish. If selenium forms an essential ingredient, it forms a sublimate of selenium, steel-gray near the assay and red at a greater distance; small, very volatile crystals of selenous acid are sometimes seen still further from the assay.

γ. **Arsenic.**—Metallic arsenic and arsenides, which contain so much arsenic, that, after the formation of basic arsenates in the tube, there remains still free arsenic, yield a crystalline sublimate of arsenous acid, very volatile, and therefore at some distance from the assay. It can be driven off by simply warming it in the tube. When there is only enough to form basic arsenates, as with speisses rich in cobalt and nickel, a distinct sublimate is not always obtained, even when the substance is powdered.

When *too large a piece of metallic* arsenic is *taken*, or an *arsenide containing* very much arsenic, *too high a heat* may *easily form* a *brownish-black* sublimate of sub-oxide of arsenic, or even metallic arsenic, and then the garlic odour of the suboxide becomes perceptible. When an easily decomposed sulphide is present, a high heat tends to produce a sublimate of yellow or red sulphide of arsenic.

δ. **Antimony.**—Metallic antimony, antimonides, sulphide of antimony, and compounds of metallic sulphides, containing sulphide of antimony, oxidize and yield white fumes, consisting at first of oxide of antimony, but changing at a high enough temperature under the influence of the air for the most part into a combination of oxide of antimony and antimonic acid. Pure oxide of antimony is volatile, and passes through the whole length of the tube as a white vapour, condensing partly on the upper side of the tube and partly escaping. It can be driven off by heating it again to redness, but a part is very liable to become more oxidized and remain behind. The combination of antimonic acid and oxide of antimony once formed, is not volatile, and condenses chiefly on the lower side of the tube as a white powder, yellowish while hot. It is mostly formed in roasting sulphide of antimony and its compounds with some metallic antimonides containing separate constituents which are easily oxidizable and evolve considerable heat in oxidizing. In presence of sulphur, sulphurous acid also forms and can be detected as usual. If the substance consists of oxide of antimony, or contains free oxide, a portion sublimes unaltered, while part is oxidized more highly and remans behind. When sulphide of lead as well as antimony is present, as in bournonite, a sublimate of oxide of antimony with antimonate of lead forms, the latter having a light yellow colour.

When sulphide of antimony, or compounds containing much of it, are very strongly heated, the white coat is likely to be spotted here and there with a reddish or brownish sulphide, which condenses in combination with the oxide. *Vide* p. 60, δ.

ε. **Tellurium** and tellurides form tellurous acid, which passes as a white vapour through the tube, and soon condenses for the most part into a sublimate, which can be fused into colourless drops by heating that part of the tube with the blowpipe, and is thus distinguished from that produced by oxidizing antimony.

ζ. **Mercury.**—Its compounds with other metals give a sublimate of metallic mercury consisting of small globules, which almost adhere together, and can be united to one drop by striking the tube and turning it slowly on its axis. Sulphide of mercury is by careful heating decomposed into sulphurous acid and mercury, the latter forming a metallic mirror above the assay. By too strong a heat part of the sulphide sublimes unchanged and forms a black sublimate close to the assay, which can be decomposed by carefully warming it. Compounds of chlorine and mercury are very volatile and sublime unaltered.

Sulphide of lead yields, besides sulphurous acid, a white sublimate of sulphate of lead, chiefly on the under side of the tube, which when strongly heated melts to yellow drops, white when cold. The assay is surrounded with fused oxide of lead, containing sulphate, which is yellow when hot and lighter on cooling. *Chloride of lead,* and substances containing it, form a sublimate which on repeated heating can only be partly volatilized, with the chlorine liberated from the residue. This consists of oxichloride of lead and fuses like tellurous acid to drops, which are, however, yellow when hot and pearl-gray to white on cooling. *Sulphide of bismuth* behaves quite like sulphide of lead. *Metallic bismuthides* yield oxide of bismuth, which condenses quite near the assay, and is fusible to brown or dark-yellow drops very different from tellurous acid. *Sulphide of molybdenum* roasts with difficulty, yielding sulphurous acid, and when powdered and heated strongly for some time, a trifling crystalline sublimate of molybdic acid.

3. EXAMINATION OF THE SUBSTANCE BEFORE THE BLOWPIPE ON CHARCOAL.

When the substance is solid and does not decrepitate, a small fragment can be used, otherwise it must be very finely pulverized. The assay is placed on the side of the coal which shows the edges of the annual rings and near the end turned toward the lamp, a slight cavity being made to receive it. A gentle O. F. is directed upon the assay, while the coal is held *horizontal and in the direction of the blowpipe flame.* The flame, being inclined upon the assay at an angle of about twenty degrees, is continued for a short time only, but until a change of colour, glowing, swelling, fusion, or the separation of some volatile substance is noticed, or in general any alteration of the substance likely to afford a clue to its character. At the moment when the blast is stopped the, presence of any volatile acids, or of sulphur, arsenic, or selenium, is tested by smelling.

Since a trifling quantity of arsenic is not thus so easily detected as sulphur or selenium, the assay is also treated with the R. F., when the arsenic odour frequently becomes very distinct. While heating the assay various other phenomena are to be noticed;—as any deflagration after the fusion, which occurs in case of nitrates, chlorates, bromates, and iodates; whether the coal is coated with volatilized bodies, far from the assay, or near it; what colour the coat has when hot and cold; whether it can be volatilized by simply warming it with the flame, or by contact with the flame, and in the latter case whether it colours the outer flame in disappearing.

An earthy substance, after being ignited thoroughly, is laid on moistened red litmus-paper, to see whether it has an alkaline reaction, which is the case with compounds of the alkaline earths with carbonic, sulphuric, and nitric acids, and of their radicals with chlorine, bromine, iodine, and fluorine.

An important means of recognizing various substances is afforded by the coats which they form on the coal at greater or less distances from the assay; but the ashes must not be mistaken for a coat, which they sometimes resemble. This can be guarded against by treating the coal previously with the O. F., to ascertain nearly the quantity and quality of the ash.

The following bodies yield characteristic coats:—

a. **Selenium** melts very easily and yields brown fumes in the O. F. and R. F., while a steel-gray coat with a feeble metallic lustre forms near the assay, and a dark-gray dull coat at a greater distance. This coat can be driven about quite easily with the O. F., and if treated with the R. F., disappears with a fine azure-blue flame. During all these operations a strong horseradish odour results from the escaping gaseous and colourless oxide.

β. **Tellurium** melts very easily, volatilizes in fumes, and coats the coal in the O. F. and R. F. with tellurous acid, at no great distance from the assay. The coat is white, but has a red or dark-yellow border, and can be driven about with the O. F., while it disappears under the R. F. with a green, or in presence of selenium, a bluish-green, flame.

γ. **Arsenic** volatilizes without fusing, and coats the coal with arsenous acid in both flames. The white coat appears grayish when thin, partly from the coal showing through and partly from a mixture of suboxide, and is at quite a distance from the assay. It can be immediately driven off by simply warming it with the flame, and if rapidly treated with the R. F., colours it pale light blue. During volatilization arsenic evolves a strong alliaceous odour, due to the formation of suboxide.

δ. **Antimony** melts very easily, coating the coal with oxide in both flames. The white coat, bluish in thin layers, is not so distant as with arsenic. It can be driven about by heating it gently with the O. F., and disappears under the R. F., tinging it pale greenish. It is not so volatile as the arsenous acid coat. When antimony is melted and heated to redness on coal and the blast stopped, it remains melted and glowing for some time, evolving dense, white fumes, which condense partly on the coal and finally surround the button with white, pearly crystals. This results from the oxidation of the metal by the air forming oxide of antimony and evolving so much heat that the fusible metal is kept fluid, until covered with crystals of oxide.

ε. **Thallium** melts very easily, forming at some distance from the assay a moderate white coat of oxide, which can be driven off by simple warming, and tinges the R. F. green. The melted button, which also tinges the flame strongly green when touched by it, remains, after setting the coal aside at rest, for some time fluid, in consequence of continuing oxidation, and occasionally a brown coat is observed very near the metal, which is perhaps peroxide.

ζ. **Lead** melts easily, *coating the coal with oxide in both flames. When warm, this is dark lemon-yellow, when cold, sulphur-yellow and bluish-white in thin layers. The yellow* coat is pure oxide of lead, and the bluish-white one, carbonate. When the coal is heated to glowing, the coat changes its place, because the oxide is reduced and the metal immediately volatilized and reoxidized. The flame is thereby coloured azure-blue.

η. **Bismuth** fuses very easily, giving a coat of oxide, which is dark orange-yellow when hot and lemon-yellow when cold, being yellowish-white in thin layers. The yellow coat is pure oxide, and the yellowish-white one, which is at the greatest distance, is carbonate with some oxide of bismuth. It can be driven about on the glowing coal like lead, but does not colour the R. F. during the operation.

θ. **Cadmium** melts very easily and burns in the O. F. with a dark-yellow flame and brown fumes, coating the coal with oxide rather near the assay. Nearest the assay the coat is thick, crystalline, and of a very dark, almost black colour, further off it is reddish-brown, and finally orange-yellow in thin layers. It can be easily reduced and driven about with either flame, but gives no coloration. Beyond the furthest limits of the coat the coal sometimes shows a variegated tarnish, ι. **Indium** fuses easily, yielding a coat which is very near the assay, dark-yellow while warm, but yellowish-white on cooling, and is driven off with difficulty by the R. F., to which it imparts a fine violet tinge.

κ. **Zinc** fuses easily and burns in the O. F. with a strongly luminous, greenish-white flame and thick white fumes, which coat the coal with oxide. The coat is rather near the assay, yellow while warm and white when cold. It becomes luminous under the O. F., but is not volatilized, because the glowing coal cannot reduce it. It is volatilized only slowly even by the R. F.

λ. **Tin** fuses easily, and in the O. F. is covered with oxide, which can be mechanically blown away; in the R. F. the metal becomes lustrous and coats the coal with oxide, which is pale yellow while warm and luminous under the O. F. On cooling it becomes white. It is so close to the assay that it borders directly upon it and cannot be volatilized by either flame, but is slowly reduced to metallic tin in the R. F.

μ. **Molybdenum**, in the powdered state, is infusible, but heated with the outer flame gradually oxidizes and coats the coal at a little distance with molybdic acid, which in many places, and particularly nearest the assay, condenses in transparent crystalline scales, but beyond in a pulverulent state. While hot the coat is yellowish, but white on cooling. On touching only its surface with the yellow flame a very fine dark blue colour of molybdate of molybdenum is formed; if, however, the heat was so great as to cause the coal to glow, the latter when cool has a dark copper-red, metallic appearance, from binoxide of molybdenum, which has been reduced from the molybdic acid and is not volatile. In the R. F. metallic molybdenum is unaltered.

ν. **Silver** fused for some time with a powerful oxidizing flame gives a slight reddish-brown coat of oxide. In combination with a little lead there is first a yellow coat of oxide of lead; but afterward, when the silver is more free from lead, a dark-red coat forms outside of the first. When a little antimony is present there results at first a white coat of oxide of antimony, which becomes red on continued blowing. In case the silver contains a little of both metals, a copious crimson coat is formed after most of the lead and antimony are volatilized. This coat is obtained sometimes in testing rich silver ores on coal.

Many substances yield coats similar to those above, and regard must be had to these in order to avoid mistakes and confusion. They are: certain sulphides, chlorides, bromides, and iodides, which yield a white coat. *Sulphides of potassium* and *sodium,* having been formed in the R. F. from the sulphates, volatilize and are oxidized, giving a white coat of neutral sulphates, which does not form, however, until all of the sulphate has sunk into the coal and been reduced. Sulphide of potassium being more volatile than sulphide of sodium, forms a thicker coat. When the coat is touched by the flame it disappears with a bluish-violet colour in case of potassa and a reddish-yellow colour in case of soda. *Sulphide of lithium* behaves similarly and colours the flame purplish-red, while the newly-discovered cæsium and rubidium sulphides probably show similar reactions. *Sulphides of lead* and *bismuth* give two coats, one more volatile and white, consisting of sulphate, and one nearer the assay, consisting of oxide of the metal in hand, which can be known by its colour both while hot and cold. The white lead coat disappears with a blue flame, and both it and the bismuth coat leave a spot of yellow oxide where the coal was made to glow. *Sulphides of antimony, zinc,* and *tin* yield only oxides, either volatile, in case of antimony, or fixed, in case of zinc and tin.

Many chlorides volatilize and yield a white coat. With *chlorides of potassium, sodium,* and *lithium* the coat is not formed until they have fused and sunk into the coal. Chloride of potassium gives the thickest coat and lithium the feeblest, which is also grayish-white and not pure white. The *chlorides of ammonium, mercury,* and *antimony,* volatilize without fusing, and those of *zinc, lead, bismuth,* and *tin,* first fuse and then give two coats; one of the chloride of the metal, white and more volitile; the other of the oxide, and less volatile. Some of these chlorides colour the R. F.: that of potassium bluish-violet, of sodium reddish-yellow, of lithium purplish-red, of lead blue: the others disappear without colouring it. *Chloride of copper* also fuses and colours the flame intense azure-blue, and on continued blowing part of the assay volatilizes with white fumes, which smell strongly of chlorine, while another part forms three coats differing in colour; that nearest the assay is dark gray, the next is dark yellow to brown, and the furthest is bluish-white. When such a coat is touched with the flame it partly changes its position with an azure-blue coloration. Among the bromides and iodides, which behave quite like the chlorides, the *bromides* and *iodides of potassium* and *sodium* deserve especial mention here. They fuse, sink into the coal, and then volatilize with white fumes, which partly form a coat rather far from the assay. This disappears under the R. F., colouring it bluish-violet in case of potassium and reddish-yellow in case of sodium.

4. EXAMINATION OF THE SUBSTANCES AS TO FUSIBILITY AND THE COLOUR WHICH THEY IMPART TO THE OUTER FLAME.

a. Testing the fusibility of substances.

Metals, metallic compounds, sulphides, or other substances which appear to contain ingredients that would attack platinum, are heated on charcoal with the R. F. or within the blue flame. A small piece is used for the assay. Most of the metals can be fused in this way, but they generally oxidize more or less and gradually volatilize; among the noble metals, gold and silver form an exception, although silver is not altogether fixed. The other noble metals, *platinum, iridium, palladium, rhodium,* and *osmium,* are infusible on coal, whether in powder, beads, or scales. Osmium is oxidized to osmic acid, however, in the O. F., and volatilizes as such; while platinum, in very fine wire, or in very thin and pointed strips, can be melted with a good O. F.

Among the other metals, the oxides of which can be reduced in the R. F., especially by adding soda or neutral oxalate of potassa, *tungsten, nickel, cobalt, iron,* and *molybdenum* are likewise infusible on coal; the latter is gradually converted into molybdic acid in a pure O. F., p. 67. Nickel and cobalt can, however, when in very thin, pointed strips, be fused to malleable buttons with the tip of the blue flame; fine iron wire likewise melts, but gives a brittle button of magnetic oxide.

The compounds of metals with arsenic are generally fusible, even in case of infusible metals. So are the metallic sulphides, but several of them are gradually volatilized, evolving sulphurous acid and coating the coal, p. 68. Among the natural sulphides, those of manganese, molybdenum, and zinc are infusible.

The fusibility of the metallic oxides must be tested with a pure O. F. Only a few of them are fusible, including the following : oxide of copper; oxide of antimony, which volatilizes after fusion; oxides of bismuth and lead, both of which are reduced to metal by the glowing coal after fusion.

When the substance is earthy, or a silicate, or in general such as will not attack platinum, and is also in a firm state, a thin splinter, or small piece having a point or sharp edge, is held in the platinum forceps with the edge in the hottest part of a pure O. F. If, however, the substance has decrepitated when tested in the matrass, a suitable fragment of the heated substance is selected. For substances which crumble to powder or cannot be divided into sharp fragments, the method proposed by Berzelius for minerals that fuse with great difficulty is employed. The substance is rubbed fine with a little water in the agate mortar, the thin paste laid on coal, dried, and heated B. B. quite strongly toward the end, until the mass lies loosely on the charcoal as a thin, coherent plate, which can be held with the platinum forceps in the O. F. Substances already in a powdered state can be similarly treated.

By heating the edge or point of the assay in a pure, sufficiently strong O. F., so that there is a slight interval between the tip of the blue flame and the assay, it will soon be seen whether the substance is fusible or not. Infusible substances retain their sharp edges unaltered, which can only be ascertained by using the magnifier: those of difficult fusibility are rounded on the edge, while the easily fusible ones melt to a bead.

Substances may be divided according to their fusibility into:

1. Such as fuse to a bead; *a*, easily; *b*, with difficulty.

2. Such as fuse only on the edge; *a,* easily; *b,* with difficulty.

3. Such as are infusible.

Von Kobell has arranged a scale by which the fusibility of the substance may be compared with that of certain minerals of known fusibility, and thus more exactly determined.

1. **Stibnite,** fusible in the flame of a candle in coarse splinters.

2. **Natrolite,** fusible in the candle flame only in fine splinters, easily fused before the blowpipe in quite coarse fragments.

3. **Almandite** or **iron alumina garnet,** infusible in the candle flame, quite fusible before the blowpipe in coarse fragments.

4. **Actinolite,** fusibility notably less than almandite and greater than orthoclase, fusible in coarse splinters.

5. **Orthoclase,** fusible in fine splinters.

6. **Bronzite,** only rounded on the edges in very fine splinters.

Splinters of the above minerals, varying in fineness, may be kept on hand for comparison; the various gradations of fusibility are expressed by decimals, thus: fusibility 2·7–2·8 denotes that the mineral is somewhat more fusible than almandite.

Some minerals infusible in the O. F. can be fused on the edges in the R. F., or even in the tip of the blue flame. Thus, *hematite* loses some of its oxygen in the R. F., and is then fusible on the edges; *magnetite* becomes more highly oxidized and is infusible in the O. F., while it can be fused in the R. F.; *siderite* when ignited in the matrass is converted into proto-sesquioxide of iron, at the expense of the carbonic acid driven off, and then behaves like magnetite; *chromite, titanic iron, franklinite,* and the *silicates of protoxide of iron* also act similarly. Substances containing oxides must therefore always be tested at first with a pure O. F., and the R. F. only used when they appear infusible, so as to ascertain whether any difference is to be perceived.

Many minerals when strongly heated alter in form and colour without fusing; some swell, like borax; others give cauliflower-like ramifications, part of them fusing after swelling up, while others remain puffed up without fusing. Some minerals fuse and froth up, giving a blebby glass filled with bubbles, which cause it to appear opaque, even when the glass is really transparent. This intumescence and bubbling generally do not appear until the temperature is sufficient to expel all the water. The cauliflower-like excrescences seem to result from a change in the manner of combination of the constituents, and in their relative position, brought about by the heat; while the tumefaction and bubbling, which occur in an already fused mass, are apparently produced by the evolution of some volatile ingredient in a gaseous state, although it does frequently appear in compounds where analysis fails to indicate the presence of any such body. It occurs particularly in the silicates of lime or an alkali with alumina.

β. *Examination of the substance as to the colour it imparts to the exterior of the blowpipe flame.*

There are many bodies which colour the outer flame more or less when heated with the tip of the blue flame. When the colour is distinct and sharply defined it serves frequently as a characteristic means of immediately recognizing the constituents of the substance. This test may frequently be combined with that for the fusibility, because it is generally made with small splinters of hard minerals, held in the platinum forceps, or in case of powdered or decrepitating substances, with a crust prepared with water, as directed on p. 70.

The splinter, or crust, being first tested in the platinum forceps with regard to its fusibility, its sharp point or edge is then brought into direct contact with the tip of the blue flame, so as to see whether the exterior pale-bluish flame is coloured. With many substances the slightly luminous envelope of the blue flame streams past the assay without change of colour, but with many other substances the outer flame is first somewhat enlarged, in consequence of a trifling amount of water or carbonic acid, and frequently coloured a feeble reddish-yellow, but this tinge disappears afterward, giving place to a very different one, which is produced by volatilizing constituents. There are also substances which produce the colour immediately, and if the assay fuses with difficulty, or not a t all, the colour frequently becomes more intense after continuing the blast longer. When the assay was fused to a bead in testing its fusibility, and no longer produces a distinct colour, a fresh piece should be used, because a fused bead does not produce so intense a colour as a fusing point or edge.

Many substances produce no colour, or only an indistinct one, although containing a constituent which has the quality of colouring the flame when free; as substances containing phosphoric acid, boracic acid, or lithia, in small quantity. The finely-powdered substance is then treated on platinum wire, with special fluxes, or after being moistened with sulphuric acid.

Easily fusible substances are tested on platinum wire, but it is always well to take only a small quantity, which can then be heated strongly, and will produce a more intense colour than a larger amount. To fix tho substance on the wire, this is heated and brought into contact with the substance, or first moistened with distilled water if the substance will not adhere to the glowing wire. Hydrous salts adhere very easily to the hot wire, but anhydrous ones only with difficulty, if at all. Metals and their combinations, sulphides of the metals and easily reducible oxides in the powdered state, must be treated on charcoal. If they are massive, a fragment the size of a hemp-seed is used, and if in powder, a quantity which would not be larger when melted. In either case the assay is laid in a quite shallow cavity on the long side of the coal, and the blue flame is directed immediately upon it, when, if the substance possesses the quality of colouring the flame, the assay will be surrounded with a distinct and more or less intensely-coloured flame. If the substance forms a coat on the coal, this can likewise be treated with the blue flame, but it is better then to employ a more spreading R. F., so as to observe distinctly the colour which the coat produces in disappearing.

All experiments on the colour of the flame, in whatever way conducted, succeed better either in a darkened room, or, if in a light room, when the operator places himself before the lamp, so that the daylight may not fall directly upon the flame, since in this way only can the exterior feebly-coloured envelope of the blue flame be most plainly seen.

Too much care cannot be exercised in these experiments, to avoid an impure flame caused by soda, since even the minutest amount of a soda salt renders the experiment useless, because soda colours more intensely than any other body. The assay must therefore be handled as little as possible with the fingers, and if pulverized in the mortar with water, care must be taken to cleanse this from any borax or soda by washing. The platinum wire should also be pure and impart no colour to the flame when tried by heating it in the point of the blue flame. Should it show a reddish-yellow colour, which may be caused by chloride of sodium from the moist fingers, this will disappear after continuing the blast some time; but if a very trifling quantity of some previously tested substance containing soda still adheres to it, the intense reddish-yellow flame will be permanent and the wire must be cleansed, either by warming it with hydrochloric acid in a test-tube and then washing it with water, or by fusing a little bisulphate of potassa on it and then shaking this off.

The colours imparted to the outer flame, by heating various bodies with the blue flame, are *yellow*, *violet*, *red*, *green*, and *blue*.

1. **Yellow.**—All soda salts fused in contact with the point of the blue flame colour the outer flame intense reddish-yellow. A large admixture of other salts, the bases of which colour the flame less intensely than soda, does not conceal this reaction. Small splinters of silicates containing soda, strongly heated or fused in the forceps, colour the flame more or less strongly, according to the proportion of soda in them.

2. **Violet.**—Potassa and most of its salts, with the exception of borate and phosphate, as well as salts of rubidium and cæsium and the compounds of indium, tinge the outer flame with a bluish-violet colour. The three latter elements being exceedingly rare, and occurring in very minute quantities, the most important colour is that produced by potassa. Even the minutest mixture of a soda salt alters this reaction, so that, although a feeble violet colour can be seen quite dis-

tinctly near the assay, the intense soda flame is apparent at a greater distance. If the amount of the soda salt rises to several per cent., the potassa reaction is entirely concealed, and in such cases the coloured flame is viewed through blue cobalt glass, or a solution of indigo, to recognize the potassa flame, as will be more particularly described under potassa. The potassa reaction is likewise concealed by lithium, unless present in very small quantities. Silicates containing considerable potassa only produce the violet flame when perfectly free from soda and lithia, and rather easily fusible on the edges.

3. **Red.**—There are three bodies which produce a red flame: *lithia*, *strontia*, and *lime*.

a. **Lithia** and its salts produce a carmine-red flame, the chloride colouring most intensely. Notable admixtures of potassa salts do not prevent the reaction, but produce at most only a violet shade; quite small quantities of soda salts, however, suffice to change it to a yellowish-red flame. With a considerable mixture of a soda salt the flame becomes only intense reddish-yellow, and cannot be distinguished from a pure soda flame. The means of distinctly recognizing lithia in such cases are given under lithia. Many of the lithia minerals show the presence of lithia more or less distinctly, as lepidolite, spodumene, and petalite. Triphylite (phosphate of lithia, iron, and manganese) fused in powder on platinum wire, produces in the outer flame a red streak of lithia, surrounded by a green envelope, which results from the phosphoric acid. The reaction cannot be readily observed in the forceps, because triphylite fuses easily. *Vide* also silicates under lithia.

β. **Strontia.**—Chloride of strontium produces immediately a crimson flame. Many strontia salts, as the carbonate, *strontianite*, and sulphate, *celestite*, when heated in the forceps, colour the flame at first pale yellowish, but afterward crimson. In presence of much baryta the strontia flame may be entirely concealed by the green baryta flame.

γ. **Lime.**—Chloride of calcium produces a red flame, less intense than chloride of strontium and mixed with yellow. Fluorite fuses at first and colours the flame yellowish, but as the fused mineral is altered to basic fluoride of calcium and becomes less fusible, the flame changes to an intense yellowish-red lime flame. Most pure calcites and compact limestones yield a pale-yellowish flame at first; but as the carbonic acid is expelled a red flame appears, which is, however, less intense than with the two salts above named. The presence of baryta prevents the reaction. Gypsum and anhydrite produce at first only a feeble yellowish flame, but afterward a slight red flame. Among the silicates *wollastonite* alone produces a feeble red lime flame.

4. **Green.**—Several bodies produce a green flame; viz., *oxide of copper, thallium, boracic acid, tellurous acid, baryta,molybdic,* and *phosphoric acids.*

α. **Oxide of Copper,** both alone and in combination with acids which themselves produce no coloration, gives an emerald-green flame, viz., carbonate and nitrate of copper. Silicates and other compounds containing oxide of copper also generally produce a very intense green, as dioptase, chrysocolla, and the same is obtained when copper forms an unessential ingredient, as in turquois and many specimens of smithsonite. Combinations of copper and iodine also cause a very intense green flame, and metallic copper melted on coal, and not entirely protected from the air, is oxidized and yields an emerald-green flame.

β. **Thallium.**—When melted on coal and touched with the tip of the blue flame, this metal is surrounded by a green flame. Its salts gives an intense green flame.

γ. **Boracic Acid.**—Both the native and artificial acid produce a yellowish-green flame, but if not quite free from soda, the green flame is mixed with more or less yellow. Several of the minerals containing boracic acid, as datolite, boracite, produce a distinctly yellowish-green flame, while others must first be finely pulverized and treated on platinum wire with sulphuric acid. Borax produces a yellow flame, on account of its soda, but if melted, pulverized, moistened with sulphuric acid, and heated, an intensely green flame is produced for a short time, which changes to yellow again as soon as the salt is decomposed or all the free sulphuric acid gone. Another and very sure means of detecting boracic by its green flame; has been proposed by Turner, and will be specially described under boracic acid.

δ. **Tellurous Acid** fuses, fumes, and colours the flame green. The tellurous acid deposited on coal during the treatment of a tellurium ore disappears with a green colour, or in presence of selenium a bluish-green colour, if treated with the blue flame.

ε. **Baryta.**—Chloride of barium yields at first a pale-green flame, which afterward becomes intense yellowish-green. The colour is finest when very little of the salt is employed. Carbonate of baryta, *witherite*, and sulphate of baryta, *barite*, also colour the flame yellowish-green, but less intensely. The reaction is not prevented by the presence of lime, as is shown by the mineral *barytocalcite*.

ζ. **Molybdic Acid**, or binoxide of molybdenum, gives a yellowish-green flame, in which the yellow predominates more than in the baryta flame; molybdic acid volatilizes meanwhile. When the edge of a thin scale of *molybdenite* is held in the flame, it at once produces a yellowish-green molybdic acid flame, without fusing.

η. **Phosphoric Acid.**—Fuchs and Erdmann have shown that phosphoric acid, phosphates, and minerals containing phosphoric acid give a bluish-green flame; some of themselves, others only after being moistened with sulphuric acid. This reaction is so sure, that with proper care, even very small quantities of phosphoric acid can be detected in minerals, when they are finely pulverized, moistened with sulphuric acid, and treated in the loop of a platinum wire. The same is true of salts which alone give no phosphoric acid reaction, in consequence of soda or some other intensely colouring ingredient. If these salts contain water, it must first be expelled by ignition or fusion on coal, and when soda is present, although the flame is quite distinctly bluish-green so long as the phosphoric acid is liberated by the sulphuric acid, yet afterward an intense soda flame succeeds. As the bluish-green flame lasts but a short time, the assay must be closely watched as soon as it is brought near the blue flame. *Pyromorphite*, owing to the oxide of lead present, gives a blue flame, the tip of which has a persistent green colour.

Ammonia and nitric acid, especially when combined, volatilize with a bluish-green flame, which is quite similar to that of phosphoric acid, although feeble; so do chloride of ammonium, etc.

5. **Blue.**—A blue flame is produced by *selenium, arsenic, lead, chloride* and *bromide of copper,* and *antimony.*

a. **Selenium** fused within the blue flame on coal volatizes with an intense azure-blue flame. The coat formed by it shows the same reaction.

β. **Arsenic.**—Metallic arsenic and arsenides of the metals which produce no coloration themselves, viz., *niccolite, smaltite,* etc., are surrounded with a blue flame when heated on coal. A very volatile coat of arsenous acid is also formed, which, if quickly touched with the blue flame, likewise disappears with a very distinct light-blue colour. Arsenates of bases which themselves do not colour the flame, viz., *annabergite, erythrite, pitticite,* etc., heated in the forceps, give an intense light-blue flame, which is also frequently produced even when the base also causes a coloured flame, as with arsenate of lime, *pharmacolite.*

γ. **Lead.**—Melted on coal metallic lead is surrounded by an azure-blue flame and yields a coat of oxide, which if driven about gives an azure-blue flame also. Most lead salts treated on platinum wire, or in the forceps, give an intense azure-blue flame.

δ. **Chloride** and **Bromide of Copper.**—The native and artificial chlorides strongly heated colour the flame intense azure-blue, but afterward green from oxide of copper. Cupriferous substances, as metallic oxides and slags, when finely pulverized and moistened with hydrochloric acid, colour the flame azure-blue for a short time. Bromide of copper similarly treated gives at first a greenish-blue flame, but afterward the green oxide of copper flame.

ε. **Antimony** melted on coal is surrounded by a scarcely perceptible greenish-blue flame; if the resulting coat of oxide is treated with the blue flame it disappears, imparting to it a greenish-blue colour.

Substances which the preceding tests have shown to be free from combustible bodies, can be at once treated with the above reagents; sulphides and arsenides of the metals, and oxides mixed with them, must be prepared in most cases by removing the sulphur and most of the arsenic, and by thoroughly oxidizing the metals. This is done by

Roasting the substance on charcoal.

Thirty to fifty milligr., more or less, as required, of the very finely pulverized substance is pressed down with the spatula or knife-blade into a thin layer, in a quite shallow cavity scraped on the coal, and is treated at first with a feeble O. F., so that the assay is touched only by the tip of the outer flame and heated to low redness. Most of the sulphur then volatilizes as sulphurous acid, the metals are oxidized, and, since sulphurous acid has a tendency to change into sulphuric acid at the expense of already formed or forming metallic oxides, these are converted partly into sulphates, and also, in presence of arsenic, into arsenates. As soon as the fumes of sulphurous acid can no longer be smelled, a feeble R. F. is employed, which reduces the sulphates and arsenates for the most part, while the arsenic is more or less completely volatilized, according to the ease or difficulty with which the arsenides yield up their arsenic. When the arsenical odour ceases, the assay is once more ignited with a feeble O. F., generally causing a slight odour of sulphurous acid, and then the assay, which should only be baked together, but not *sintered*, and much less fused, is turned with the spatula and the other side treated alternately with the O. F. and R. F. After roasting this side, the coherent mass is powdered in the agate mortar, and since it is not free from disseminated sulphates and arsenates, or may even, if not carefully enough roasted, still contain a trifling amount of sulphides and arsenides, it is replaced on the coal and again roasted on both sides. Sulphur is frequently more easy to expel than arsenic, but there are metallic sulphides which can be converted for the most part only into sulphates, as sulphide of lead; it is also not very easy to convert sulphide of copper into oxide quite free from sulphate, by roasting, although continued heating alters the sulphate into tolerably pure oxide. By mixing the roasted assay in the mortar with an equal volume of carbonate of ammonia and again feebly igniting the mixture on coal in the O. F., sulphate of ammonia volatilizes and leaves the oxide of copper free from sulphuric acid. Arsenic often remains obstinately combined as acid with certain oxides, especially protoxides of nickel and cobalt. Antimony volatilizes partly at the outset as oxide, while the remainder is converted into a fixed combination of oxide of antimony and antimonic acid. If the substance contains much sulphide of antimony, lead, or any other easily fusible sulphide, and is liable to *sinter*, as with tetrahedrite, bournonite, etc., it is well first to expel the volatile sulphides by fusion on coal, recognizing them by the coats formed, and to roast only the residue, after again pulverizing it. To avoid the escape of arsenical fumes, substances containing much arsenic may first be ignited in an open glass tube, when the greater part of it sublimes as arsenous acid and some of the sulphur volatilizes as sulphurous acid, after which the assay is roasted on coal.

A well-roasted assay must give no odour of arsenic or sulphurous acid while glowing, must have a dull appearance, and must admit of eing easily crushed to a very fine powder; otherwise it must be pulverized and further roasted.

When treating selenides, tellurides, or antimonides containing little or no sulphur, and which have been recognized as such by the preceding tests, it is seldom necessary to roast them, because in most cases the selenium and volatile metals are volatilized by fusing them alone on coal for some time, and the fixed metals can then be very easily recognized by fusion with fluxes in the O. F. Telluride of silver forms an exception, inasmuch as it yields only a part of its tellurium, and a silver button free from tellurium cannot be otained in either flame.

I. EXAMINATION OF SUBSTANCES WITH BORAX.

This test is made either on platinum wire or on coal. Earths and metallic oxides are generally treated first on wire in the O. F., and then in the R. F., on wire or on coal; roasted sulphides and arsenides apparently free from arsenate of cobalt and nickel are likewise so treated; but substances containing much of those arsenates are treated at once on coal. The part which the borax plays as reagent has been explained.

The testing of substances with borax on platinum wire is performed as follows: The heated loop is dipped in borax, and the adhering salt melted to a glass in the O. F., repeating the operation until the bead corresponds to the size of the loop. The bead must be *quite colourless*, both when hot and cold; if otherwise, it must be removed by heating it strongly, and then shaking it off into a small porcelain vessel, by striking the hand holding it smartly upon the table. The success of the operation depends upon the rapidity of the manipulation and the firmness with which the wire is held in the hand.

The substance may then be attached to the cold, moistened borax bead, or at once caused to adhere to it while melted, and is in either case fused with it in the O. F. It must now be observed whether the body dissolves with ease or difficulty, with or without effervescence, whether the bead with the dissolved substance is coloured when held against the daylight, and whether this colour remains the same, or becomes lighter on cooling, as well as whether the bead remains clear, or becomes opaque when cold.

Some bodies give a clear glass up to a certain degree of saturation, which also remains clear when cold, but by gently heating it, especially by a quick intermitting blast, the bead becomes opaque, milk-white, or opalescent, and sometimes coloured. This operation is called *flaming*, and generally produces a result only with such bodies as yield a glass, which, after perfect saturation, is transparent while fluid, and becomes enamel-like of itself at the moment of solidification. This is the case with the alkaline earths, yttria, glucina, zirconia, oxides of cerium, tantalic and titanic acids, etc. It does not occur with some other substances, viz., silicic acid, alumina, oxides of iron, manganese, etc.; while the presence of silicic acid even prevents the phenomenon with bodies which alone give an enamel-like bead with borax on cooling. This is shown by their silicates, which give a clear glass when fully saturated, and the glass only becomes opaque when supersaturated and cold. When a borax bead shows the above phenomenon, it is said that the bead *becomes opaque by flaming*. The cause of the phenomenon lies in the imperfect fusion produced by the flaming, which causes a partial separation of the base from the basic borate; while in case of a supersaturated glass, the base is partially separated on cooling.

When treating a substance containing much of a colouring oxide, or several such oxides, but little should be dissolved at once, in order to avoid too dark a glass, the colour of which could not be recognized. When the glass is too darkly coloured, it is pressed out while soft, or, if this is not enough, some of it must then be broken off and the residue fused with fresh borax.

The colour of a bead may be examined with or without a magnifier, and it must be borne in mind that the colour with many substances is different when hot and cold.

The substance having been dissolved in the O. F., the glass is treated in the R. F., but with such a blast that no soot shall be deposited upon it. When metallic oxides or acids are present, which are reduced from borax only with difficulty, or not at all, viz., sesquioxides of cerium, manganese, iron, uranium and chromium, protoxide of cobalt, titanic and tungstic acids, etc., the bead may be treated at once on the platinum wire; but if there are easily reducible oxides present, as those of zinc, nickel, cadmium, lead, etc., the wire would be injured, and the bead must therefore be shaken from it and reduced on charcoal. The bead is placed in a cavity on coal and treated with a pure R. F. that deposits no soot. After blowing one or two minutes, the glass is pinched with the forceps and drawn out a little, so that its colour can be plainly seen. If the dissolved oxide was combined with a notable amount of sulphuric acid, sulphide of sodium is liable to form, which colours the glass yellowish-red, especially when slowly cooled, and a false result may be obtained unless this is borne in mind. If the glass contains easily reducible oxides of volatile metals, a coat of these oxides is formed on the coal, as with glass containing much oxide of antimony, zinc, indium, cadmium, bismuth, or lead.

In some cases a small bit of pure tin, as large as a pin-head, is placed beside the bead, and both are fused for a moment in the R. F. The tin has a great affinity for oxygen, and absorbs it partially from the metallic oxides in the glass, dissolving itself to a colourless bead, while the oxides are reduced to the lowest stage of oxidation, and produce a distinct colour, which frequently appears only after they are quite cold.

When copper or nickel is to be perfectly reduced from a borax bead containing few or no other easily reducible oxides, some metallic lead may be with advantage added to the bead on coal; the metal distributed through the bead then unites into one button with the lead, and the glass can afterward be further tested for non-reducible oxides on platinum wire.

Many metallic arsenides, as *niccolite*, *smaltite*, *cobalt* and *lead speisses*, etc., in which arsenide of nickel or cobalt forms a chief ingredient, can be immediately treated with borax on coal without roasting, since they generally fuse easily. The method of proceeding will be given under nickel and iron.

The following table, similar to those arranged by H. Rose and Scheerer, gives a convenient survey of the colours imparted to borax by the metallic oxides and acids, in both the oxidizing and the reducing flames :—

TABLE I. The Metallic Oxides and Acids arranged with reference to the Colours which they impart to the Borax Bead.

WITH BORAX IN THE OXIDIZING FLAME PRODUCE:

a. Colourless Beads.

Hot and cold:	Silica, Alumina, Binoxide of Tin. Baryta, Strontia, Lime, Magnesia, Glucina, Yttria, Zirconia, Thoria; Oxides of Lanthanum and Silver; Tantalic, Niobic, Hyponiobic, and Tellurous Acids.	When highly saturated opaque (white) by flaming.
	Titanic, Tungstic, and Molybdic Acids; Oxides of Indium, Zinc, Cadmium, Lead, Bismuth, and Antimony.	When feebly saturated.

b. Yellow Beads.

Hot:	Titanic Acid, Tungstic Acid, Molybdic Acid; Oxides of Zinc and Cadmium.	When highly saturated; on cooling colourless; and opaque by flaming.
	Oxides of Lead, Bismuth, and Antimony.	When highly saturated; colourless on cooling.
	Sesquioxides of Cerium, Iron, and Uranium.	When feebly saturated; *more or less colourless on cooling.*
	Sesquioxide of Chromium (feebly saturated); when cold, yellowish-green.	
	Vanadic Acid; *when cold, greenish-yellow.*	

c. Red to Brown Beads.

Hot:	Sesquioxide of Cerium; *on cooling,* yellow, enamel-like by flaming.
	" " Didymium (rose-coloured); the same on cooling.
	" " Iron; *on cooling,* yellow.
	" " Uranium; on cooling, yellow; enamel-yellow by flaming.
	" " Chromium; on cooling, yellowish-green.
	" " Iron containing Manganese; on cooling, yellowish-red.
Cold:	Protoxide of Nickel (reddish-brown to brown); violet while hot.
	Sesquioxide of Manganese (violet-red); violet while hot.
	Protoxide of Nickel containing Cobalt (with little Cobalt, violet-brown); violet while hot.

d. Violet Beads (amethyst-coloured).

Hot:	Protoxide of Nickel; on cooling, reddish-brown to brown.
	Sesquioxide of Manganese; on cooling, red inclining to violet.
	Protoxide of Nickel containing Cobalt; when cold, inclining to brownish. With much Cobalt, violet when cold also.
	Protoxide of Cobalt containing Manganese; the same on cooling.

e. Blue Beads.

Hot:	Protoxide of Cobalt; the same on cooling.	
Cold:	Oxide of Copper (when highly saturated, greenish-blue); green when hot.	

f. Green Beads.

Hot:	Oxide of Copper; blue when cold (greenish-blue if highly saturated).	
	Sesquioxide of Iron containing Cobalt or Copper. Ozide of Copper containing Iron or Nickel.	The green colour changes on cooling, according to the saturation, as well as the proportions in which the oxides are present, to light-green, blue or yellow.
Cold:	Sesquioxide of Chromium (yellowish-green); yellow to red while hot.	

WITH BORAX, IN THE REDUCING FLAME, PRODUCE:

a. Colourless Beads.

Hot and cold:	Silica, Alumina, Binoxide of Tin.	
	Baryta, Strontia, Lime, Magnesia, Glucina, Yttria, Zirconia, Thoria; Oxides of Lanthanum and Cerium; Tantalic Acid.	When highly saturated become opaque by flaming.
	Oxide of Indium, Sesquioxide of Manganese. (With the latter the glass is liable to assume a feeble rose-colour on cooling.)	
	Niobic and Hyponiobic Acids; when feebly saturated.	
	Oxides of Silver, Zinc, Cadmium, Lead, Bismuth, Antimony, Nickel, Tellurous Acid.	After long blowing. (After a short blast, gray.)
Hot:	Oxide of Copper; if highly saturated, opaque red on cooling.	

b. Yellow to Brown Beads.

Hot:	Titanic Acid (yellow to brown); when highly saturated, enamel-blue by flaming.
	Tungstic Acid (yellow to dark); when cold, brownish.
	Molybdic Acid (brown to opaque). (Separated binoxide of Molybdenum is seen in the black bead if pressed flat.)
	Vanadic Acid (brownish); chrome-green when cold.

c. Blue Beads.

Hot:	Protoxide of Cobalt; the same when cold.

d. Green Beads.

Hot and cold:	Sesquioxide of Iron (yellowish- or bottle-green); especially when cold.
	Sesquioxide of Uranium (yellowish-green); when highly saturated becomes black by flaming.
	Sesquioxide of Chromium (light to dark emerald-green, according to the degree of saturation).
Cold:	Vanadic Acid (chrome-green); brownish while hot.

e. Gray and Cloudy Beads; the cloudiness frequently appearing distinctly during the blast.

Cold: Oxides of Silver, Zinc, Cadmium, Lead, Bismuth, Antimony, Nickel, Tellurous Acid. — With a short blast. (With a longer blast, colourless.)
Niobic and Hyponiobic Acids, when highly saturated.

f. Red Beads.

Cold: Sesquioxide of Didymium (rose-coloured), Oxide of Copper (opaque), when highly saturated; colourless while hot.

2. EXAMINATION OF THE SUBSTANCE WITH SALT OF PHOSPHORUS.

This test is likewise made partly on platinum wire and partly on coal. The action of the S. Ph. has been already explained. The S. Ph. must be melted on the wire only *gradually in small portions, since it boils violently while the water of crystallization and the ammonia are passing off, and if a sufficient amount for a test were fused at once, it would* seldom all remain on the wire. On coal the whole amount may be fused at once. The points to be observed in testing substances with borax hold good for S. Ph. as well. Silicic acid being very slightly dissolved in S. Ph., the silicates can be very easily recognized by means of it; the bases dissolve, while most of the silica separates and floats about in the fused glass in the form of a gelatinous skeleton. Many silicates, however, yield a glass which, indeed, appears clear while hot, but on cooling is more or less opalescent, and the glass must then be examined for separated silica with the magnifier while still hot. Silicates of bases which are themselves soluble with difficulty in S. Ph., as alumina and zirconia, are decomposed by the salt with great difficulty, or not at all, and this is chiefly perceptible with zirconia.

The colours produced in the S. Ph. beads are generally different from those produced in borax by the same substances, as may also be seen by comparing the following summary with that given on pp. 84–86.

TABLE II. The Metallic Oxides and Acids arranged with reference to the Colours which they impart to the Salt of Phosphorus Bead.

WITH SALT OF PHOSPHORUS, IN THE OXIDIZING FLAME, PRODUCE:

a. Colourless Beads.

Hot and cold:	Silica (very slightly soluble).	
	Alumina, Binoxide of Tin (soluble with difficulty).	
	Baryta, Strontia, Lime, Magnesia, Glucina, Yttria, Zirconia, Thoria, Oxide of Lanthanum, Tellurous Acid.	When highly saturated become opaque (white) by flaming.
	Tantalic, Niobic, Hyponiobic, Titanic, and Tungstic Acids; Oxides of Zinc, Cadmium, Indium, Lead, Bismuth, and Antimony.	When not too highly saturated. (When highly saturated, yellowish to yellow; and colourless on cooling.)

b. Yellow Beads.

Hot:	Tantalic, Niobic, Hyponiobic, Titanic, and Tungstic Acids; Oxides of Zinc, Cadmium, Lead, Bismuth, and Antimony.	When highly saturated; but colourless on cooling.
	Oxide of Silver (yellowish); opalescent on cooling.	
	Sesquioxide of Iron, Sesquioxide of Cerium.	When feebly saturated, colourless on cooling. If highly saturated, red while hot, and yellow when cold.
	Sesquioxide of Uranium; yellowish-green when cold.	
	Vanadic Acid (dark yellow); lighter when cold.	
Hot:	Protoxide of Nickel; reddish while hot.	

c. Red Beads.

Hot:	Sesquioxides of Cerium and Iron. If highly saturated, yellow on cooling.
	" " Didymium (rose-coloured when highly saturated)
	Protoxide of Nickel (reddish); yellow when cold.
	Sesquioxide of Chromium (reddish); emerald-green when cold.

d. Violet Beads (amethyst-coloured).

Hot:	Sesquioxide of Manganese (brownish-violet); on cooling, light reddish-violet.

e. Blue Beads.

Hot:	Protoxide of Cobalt; the same on cooling.
Cold:	Oxide of Copper (greenish-blue if highly saturated); green while hot.

f. Green Beads.

Hot:	Oxide of Copper; blue when cold (greenish-blue if highly saturated).	
	Molybdic Acid (yellowish-green); lighter on cooling.	
	Sesquioxide of Iron containing Cobalt or Copper; Oxide of Copper containing Iron or Nickel.	The green colour changes on cooling, according to the saturation, as well as the proportions in which the oxides are present, to light-green, blue, or yellow.
Cold:	Sesquioxide of Uranium (yellowish-green); reddish while hot.	
	" " Chromium (emerald-green); yellow while hot.	

WITH SALT OF PHOSPHORUS, IN REDUCING FLAME, PRODUCE:

a. Colourless Beads.

Hot and cold:	Silica (very slightly soluble).	
	Alumina, Binoxide of Tin (soluble with difficulty).	
	Baryta, Strontia, Lime, Magnesia, Glucina, Yttria, Zirconia, Thoria, Oxide of Lanthanum.	When highly saturated, opaque (white) by flaming.
	Sesquioxides of Cerium, Didymium, Manganese.	
	Tantalic Acid; Oxides of Silver, Zinc, Cadmium, Indium, Lead, Bismuth, and Antimony; Tellurous Acid.	By long blowing. (Otherwise gray.)
	Protoxide of Nickel (particularly on charcoal).	

b. Yellow to Red Beads.

Hot:	Sesquioxide of Iron (yellow to red); on cooling, at first greenish, then reddish.	
	Titanic Acid (yellow); violet on cooling.	
	Hyponiobic Acid (violet-brown); particularly on coal.	
	Vanadic Acid (brownish); when cold, chrome-green.	
	Titanic Acid containing Iron. Tungstic " " "	(Yellow); when cold, brownish-red (blood-red).
	Niobic " " " Hyponiobic Acid " "	(Brownish-red); when cold, dark yellow.

c. Violet Beads (amethyst-coloured).

Cold:	Niobic Acid (highly saturated); pale dirty-blue while hot.
	Titanic Acid (even when moderately saturated); yellow while hot.

d. Blue Beads.

Cold:	Protoxide of Cobalt; also while hot.
	Tungstic Acid; brownish while hot.
	Niobic Acid (very highly saturated); dirty-blue while hot.

e. Green Beads.

Cold:	Sesquioxide of Uranium; less fine while hot. Molybdic Acid; dirty-green while hot. Vanadic Acid; brownish while hot. Sesquioxide of Chromium; reddish while hot.

f. Gray and Cloudy Beads; the cloudiness frequently appearing distinctly during the blast.

Cold:	Oxides of Silver, Zinc, Cadmium, Indium, Lead, Bismuth, Antimony, Nickel; Tellurous Acid.	Most readily on coal and with tin. Colourless after prolonged blowing.

g. Red Beads.

Cold:	Sesquioxide of Didymium (rose-coloured). Oxide of Copper (opaque), when highly saturated, or with tin on charcoal.

3. EXAMINATION OF SUBSTANCE WITH CARBONATE OF SODA.

The soda is employed, either simply to fuse the substance together with it, or to effect the reduction of metallic oxides present, which latter result can generally be more perfectly accomplished with its aid than by the reducing flame alone.

a. The fusibility of the substance with soda.

A large number of bodies combine with soda at a high temperature, some yielding fusible, and some yielding infusible compounds. There are only a few which yield fusible compounds; they are chiefly:—silicic, titanic, tungstic, molybdic, tantalic, vanadic, and the acids of niobium. Fused on charcoal, silicic and titanic acids unite with soda, effervescing and yielding a clear bead. When there is no excess of soda, the silicate remains clear on cooling, but the titanate becomes crystalline and opaque. Tungstic and the other acids likewise unite with soda with effervescence, but the compound sinks into the coal. Baryta and strontia salts likewise give fusible compounds with soda, and these also sink into the coal, while most lime salts, although fusing with the soda, are decomposed, even when their acids are more powerful than carbonic acid, and the soda salt sinks into the coal, leaving the lime behind.

The powdered substance to be tested is mixed with the soda in the left hand, and the moistened mixture, spread in a shallow cavity on coal, is heated at first gently to drive off all the water, and then as strongly as possible. It is generally advisable to add the soda in small portions, so as to note clearly the changes produced by adding constantly increasing amounts, which are spread moist upon the fused mass. Many silicates which are themselves fusible with difficulty, while their bases are infusible, melt with a little soda to a clear glass; but with more soda they form a slag-like or infusible mass. When the assay is not soluble in soda, but is decomposed by it, it may be seen to gradually swell and alter in appearance, while it does not fuse to a bead with the soda in whatever proportion it is added. When the substance is neither dissolved nor decomposed by soda, the latter sinks into the coal and leaves the assay unaltered.

An assay soluble in soda and free from colouring oxides, but containing *sulphuric acid* or *sulphur*, yields a glass which on cooling is yellow, or red to yellowish-brown, from the *formation of sulphide of sodium, according as there was little or much sulphur present. The spread-out mass obtained by fusing sulphates on coal with soda generally has the same colour, and when the mass, which has partially or wholly sunk into the coal, is cut out, laid on silver foil, and moistened thoroughly with water, it forms a black or dark brown spot of sulphide of silver.* This reaction is frequently used in testing a substance for sulphuric acid.

Substances containing *manganese*, even in very trifling quantity, when powdered and fused with soda on platinum foil in the O. F., yield manganate of soda, which spreads over the foil, and assumes a bluish-green colour on cooling.

When the examination of the substance by itself has caused the presence of salts of *ammonia* or *mercury* to be suspected, some of it is powdered, mixed with previously-dried soda, and heated in a matrass over the spirit-lamp; the salts are decomposed, and in the former case carbonate of ammonia, recognized by the smell and by red litmus-paper, is liberated, while in the latter case the mercury collects in drops or forms a gray film.

When silicates are fused with soda, they yield silicic acid to it, and easily fusible silicates are formed, containing a low proportion of silica. Upon adding more soda the weaker bases are separated, and the mass becomes infusible. When the oxygen of the silicic acid is at least double that of the base, the addition of just the right amount of soda forms a clear glass, which remains clear on cooling, provided the resulting double silicate is fusible. When, however, the oxygen of the acid is just equal to that of the base, the assay is indeed generally decomposed by soda with effervescence, but cannot be fused to a clear glass, because the resulting double silicate is too infusible. Fusible silicates of infusible bases yield with a little soda a clear glass; with more, an opaque glass; and with still more, are infusible, because their bases are separated by the soda.

The Oxidized Minerals arranged according to their fusibility and behaviour with Carbonate of Soda.

A. MINERALS FUSIBLE TO A BEAD.

a. With soda yield a fluid bead:

Acmite,
Allanite (Cerine), *A*[*]
Axinite, *A*,
Boracite, *A*,
Borax, *A*,
Botryolite, *A*,
Crocidolite,
Cronstedtite,
Datolite, *A*,
Elæolite, *A*,
Eudialyte,
Garnet,
Helvite,
Hydroboracite, *A*,
Ilvaite,
Labradorite,
Lapis Lazuli,
Mica, from primitive limestone,
Oligoclase,
Pyrosmalite,
Sassolite, *A*,
Scapolite, *A*,
Sodalite, from Greenland,
Spodumene,
Talc, black,
The Zeolites, *A*.

b. With a little soda a bead, with more a slaggy mass:

Amblygonite,
Fluorite,
Garnet, manganiferous,
Manganese, black silicate of (hydrous tephroite?)
Okenite,
Orthite, *A*,
Pectolite,
Pyrorthite,
Rhodonite, *A*,
Sodalite,
Sordavalite,
Vesuvianite, *A*.

c. With soda only a slag:

Amphodelite,
Autunite,
Brevicite,
Fahlunite,
Haüynite,
Heterosite,
Iron, phosphates of sesquioxide,
Pharmacolite,
Pharmacosiderite,
Polyhalite,
Pyrargillite,
Pyrope,
Saponite,
Scorodite,
Tourmaline (potash) black,
Triphylite,
Wolframite.

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[*] *A*, denotes that the mineral fuses with intumescence, effervescence, &c.

d. Sink with soda into the charcoal:

Celestite,
Witherite.

e. Fuse with soda at first more or less perfectly to a clear mass, but are decomposed by a sufficient quantity of soda, and leave behind an infusible crust, while the soda salt sinks into the coal:

Anhydrite,
Cryolite,
Gay-Lussite,
Glauberite,
Gypsum,
Polyhalite.

f. Yield with soda a reguline metal:

Minerals consisting of reducible metallic oxides and their reducible salts.

B. MINERALS WHICH FUSE ONLY ON THE EDGES.

a. With soda yield a fluid bead:

Albite,
Anorthite,
Emerald (beryl).
Euclase, *A*,
Nephelite,
Orthoclase,
Petalite,
Sodalite, from Vesuvius,
Steatite,
Titanite, *A*,
Turquois.

b. With little soda a fluid bead, with more a slaggy mass:

Enstatite,
Epidote, *A*,
Hypersthene,
Wollastonite,
Zoisite, *A*.

c. Yield with soda only a slag:

Carpholite,
Dichroite (iolite), blue,
Lazulite, *A*,
Mica, *A*, from granite,
Pimelite,
Pinite,
Plumbogummite, *A*,
Pyrochlore,
Scheelite,
Serpentine,
Tourmaline (soda), green, *A*.

d. With soda goes into the coal:

Barite.

e. Fuse or only swell up with soda, but are decomposed by a sufficient amount of soda, leaving an infusible crust, while the soda salt sinks into the coal:

Apatite (swells up).
Barite (calciferous), fuses.

C. INFUSIBLE MINERALS.

a. Give with soda a fluid bead:

Agalmatolite,
Dioptase,
Fire-clay,
Hisingerite,
Leucite,
Pyrophyllite, *A*,
Quartz,
Rutile,
Sideroschisolite,
Wolchonskoite.

b. With a little soda a bead, with more a slaggy mass:

Cerite,
Chrysolite,
Gadolinite, *A*,
Phenacite,
Picrosmine,
Talc,
Tourmaline, *A* (lithia).

c. Yield with soda only a slag:

Æschynite, *A*,
Allophane,
Aluminite,
Alunite;
Alunogen, *A*,
Andalusite,
Brucite,
Calamine (a zinc coat),
Cassiterite, with much soda metallic tin,
Chloritoid,
Chromite;
Chrome Ochre,
Chrysoberyl,
Cyanite,
Diaspore,
Fluocerine,
Gahnite (a zinc coat),
Gehlenite,
Gibbsite,
Iron, sesquioxide and its sulphates,
Manganese, oxides,
Œrstedite,
Ouvarovite,
Polymignite,
Spinel,
Staurolite,
Tantalite,
Thorite,
Titanic Iron,
Topaz,
Wörthite,
Xenotime,
Yttrocerite,[1]
Yttrotantalite,
Zircon.

d. Fuse or only swell up with soda, but are decomposed by a sufficient amount, and the soda sinks into the coal, leaving an infusible crust:

Alum (kalinite), dehydrated,
Aragonite,
Barytocalcite,
Calcite,
Dolomite,
Epsomite, dehydrated,
Magnesite,
Wavellite, *A*.

e. Sinks with the soda into the coal:

Strontianite, *A*.

β. *Reduction of metallic oxides with carbonate of soda.*

Many oxides can be reduced on coal in the R. F. without soda, but when mixed or combined with non-reducible bodies it is not only difficult, but sometimes quite impossible so to reduce them that their presence may be at once ascertained; by the addition of soda this can, however, be very perfectly accomplished. There are also metallic oxides which can be reduced perfectly with soda, but not without it. The easy reduction effected by soda is to be ascribed as well to the formation in the R. F. of cyanide of sodium, which absorbs oxygen with great eagerness to form cyanate of soda, as also, without doubt, to the fact that at a sufficiently high temperature the salt sinks into the coal, while its carbonic acid and part of its oxygen are converted by the charcoal into carbonic oxide, which, in connection with the gaseous sodium that escapes, exerts a reducing action on the metallic oxides.

The best way to perform this reduction is to mix the pulverized assay with moistened soda in the left hand, spread the paste on coal, and treat it with a good R. F. for not too short a time. It is sometimes difficult at once to recognize the separated metal, and then the whole of the coal which is permeated with the soda at the spot where the reduction was effected, must be cut out, triturated with water in the mortar, and the coal, etc., washed away carefully until only metallic particles remain. When even a trifling amount of a reducible oxide was present, there will be small, flattened, lustrous metallic scales at the bottom of the mortar, or, in case the metal was difficultly fusible, and not soft, a metallic powder. This residue should be examined with the glass, and under water with the magnet, and if necessary, also tested on coal with borax and S. Ph., in case a mixture of several metals was obtained.

In order to transfer the fine particles of metal to the coal, for the purpose of examination, they are wiped from the mortar with a bit of filter-paper, which is then rolled up and burned on the coal. If there is a very trifling quantity of metal, the borax or salt of phosphorus necessary for the examination is wrapped in the paper at the same time. When there are several reducible oxides present, they are generally obtained as an alloy. A little borax should be added to the soda when treating tantalates and slags, to reduce the trifling amount of oxide of tin which is often present. The compounds of tantalic or silicic acid are more easily dissolved by adding borax, which also prevents the reduction of iron that would otherwise alloy with the tin.

The metals reducible as above with soda are, besides the noble metals: molybdenum, tungsten, antimony, tellurium, copper, bismuth, tin, lead, zinc, indium, cadmium, nickel, cobalt, and iron. Arsenic and quicksilver are reduced, but volatilize immediately, and can only be obtained in the metallic state in the glass tube or matrass. If the assay contained arsenate of nickel or cobalt, a quite fusible button is always obtained, which is rendered brittle by the considerable amount of arsenic in it. When, in addition to oxide of copper, there is an oxide of antimony or tin present, an easily fusible, but brittle, alloy of copper and antimony or tin is obtained.

Neutral oxalate of potassa, or *cyanide of potassium,* may with advantage be substituted for soda when treating oxides of difficult reducibility, as they have a much greater reducing power. Even a feeble R. F. suffices to reduce oxides of tin, iron, cobalt, etc., immediately with these reagents, while soda would require a long-continued and strong blast. This is due to the fact that the oxalate, heated to incipient glowing, evolves carbonic oxide copiously, and this has a powerful reducing action, while the simultaneously formed carbonic acid, which has been absorbed by the potassa liberated from the decomposed salt, is likewise converted into carbonic oxide by the action of the coal at a higher temperature; the cyanide of potassium, on the other hand, takes oxygen directly from the metallic oxides at a low temperature, and is converted into cyanate of potassa, so far as this oxygen suffices. The cyanide, however, has the disadvantage of spreading out over the coal at once, and thus scattering the reduced metal; while the oxalate, although also easily fused, spreads less, and the metallic particles can be more readily collected to larger buttons. Both of these reducing fluxes are also preferable to soda when the reduction must be performed in a matrass, as for detecting a trifling amount of arsenic, the special directions for which will be given under the examination for arsenic.

4. EXAMINATION OF SUBSTANCES WITH COBALT SOLUTION.

This test can only be employed for substances which have a nearly or quite white colour after ignition in the O. F. If the substance is not very dense, and will absorb the solution, a fragment of it is moistened with the solution, and gradually ignited quite strongly with the O. F., being held in the forceps. Friable substances are mixed with the solution and spread on coal. In testing coats, a few drops of the solution are put on the coat, and it is then cautiously ignited, so as not to blow away the thin layer of oxide. Crystalline substances, and such as are too dense to absorb the solution, must be pulverized, mixed with a little water, spread on coal, and dried. The crust is then moistened with cobalt solution, and gradually heated to a feeble glow in the O. F. If coherent, the crust may be removed from the coal and held in the forceps. The colour imparted to the assay must always be examined by daylight and when the assay is quite cool. The colours frequently seen when the substance is moistened with the solution, or on commencing to heat it, such as blue, red, black, proceed, it is true, from decomposition of the solution, but are by no means to be regarded as indications of substances sought.

The quantity of solution required depends upon its concentration; a few experiments will show how much should be employed to secure a distinct reaction. A very dilute solution always yields the best results, as a too concentrated one is liable to turn the ignited assay gray or black.

The following colours are assumed by some earths and metallic oxides and acids on being moistened with cobalt solution and ignited:

a. *Brownish-red*, baryta;
b. *Flesh-colour*, magnesia, tantalic acid;
c. *Violet*, zirconia (dirty-violet), phosphate and arsenate of magnesia (fuse at the same time).
d. *Blue*, alumina, silica;
e. *Green*, oxide of zinc (yellowish-green), of tin (bluish-green), titanic acid (yellowish-green), hyponiobic acid (dirty-green), antimonic acid (dirty dark-green).
f. *Gray*, strontia, lime, glucina (bluish-gray), niobic acid.

Only a few of these colorations are of use in recognizing bodies, especially those of *alumina*, *magnesia*, *zinc*, and *tin*. The blue of alumina must not be confounded with the blue produced by many silicates, which is due to silicate of cobalt. This almost always appears fused on careful examination, while the blue of alumina is dull; the former also only appears with a high temperature, and it is therefore well, if the substance after ignition with the solution shows no blue colour, not to heat it too much, so as to fuse it. On the other hand, in testing for magnesia, the ignited substance may have assumed only a very feeble rose-colour, and it can then be more strongly treated, even to fusion, if possible, since the red colour will not only remain, but also become more distinct if magnesia is present.

The alumina and magnesia reactions are prevented by the presence of coloured metallic oxides, which generally produce a grey or black mass, unless present in too small quantity.

The methods of using the other qualitative reagents will be described under the special examinations.

TABLES

SHOWING THE BEHAVIOUR OF THE

ALKALIES, EARTHS, AND METALLIC OXIDES, ALONE, AND WITH REAGENTS, BEFORE THE BLOWPIPE.

ALKALIES.	ALONE ON PLATINUM WIRE.	ALONE ON PLATINUM FOIL.	REMARKS.
1. POTASSA.*	Colours the flame violet; a very little soda prevents the reaction.	0	In solution, colour red litmus-paper blue.
2. SODA.	Colours the flame strongly reddish-yellow, even when very much potassa is present.	0	
3. LITHIA.	Colours the flame carmine-red, even when more potassa than lithia is present; but a little soda alters the flame to yellowish-red or reddish-yellow.	When fused turns the foil about it dark yellow; on washing and igniting the foil this disappears, but the foil loses its polish and appears dull, especially when heated to redness.	
4. AMMONIA.	In combination with nitric and sulphuric acids and chlorine gives a pale greenish flame.	0	Recognized by its odour; also colours red litmus-paper blue.

EARTHS.	ALONE ON CHARCOAL AND IN THE FORCEPS.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA ON CHARCOAL.	WITH COBALT SOLUTION IN THE O. F.
1. BARYTA.	The hydrate fuses, boils, swells, becomes fixed on the surface, and then sinks into the coal with violent ebullition. The carbonate fuses to a clear glass easily, and becomes enamel-white on cooling. On repeated fusion it becomes caustic and is absorbed. Colours the flame yellowish-green.	The carbonate dissolves to a clear glass with effervescence, and can be flamed enamel-white with a certain amount; with more it becomes enamel-white of itself on cooling.	As with borax.	Fuses with it and is absorbed by the charcoal.	Fusible to a brownish-red bead, which loses colour on cooling, and soon crumbles to a light-brown powder in the air. With very dilute solution the bead only appears pale brown.

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* The oxides of *rubidium*, Rb, and *cæsium*, Cs, show quite similar reactions.

2. STRONTIA.	The hydrate behaves like baryta. The carbonate fuses only on the edges, ramifying like cauliflower; the projections are luminous, tinge the R. F. red, and have an alkaline reaction. In the forceps, colours the flame crimson.	Like baryta.	Like baryta.	Sr is insoluble. $\dot{Sr}\ddot{C}$ fuses with an equal amount of soda to a clear glass, milk-white on cooling. More strongly heated the glass boils and the caustic earth is absorbed. If more is added, it does not dissolve but becomes caustic and goes into the coal.	Sinters and assumes a black or dark grey colour.
3. LIME.	$\dot{Ca}$ is unaltered. Ca $\ddot{C}$ becomes caustic, whiter, more luminous, then alkaline, and crumbles to powder if moistened. In the forceps, colours the flame feebly red.	Dissolves easily. The clear glass can be flamed opaque. $\dot{Ca}\ddot{C}$ dissolves with effervescence. With more the glass becomes cloudy and crystalline on cooling, but never so milk-white as with baryta or strontia.	Dissolves largely (with effervescence, carbonate) to a clear glass, that can be flamed opaque, when rather saturated. Perfectly saturated the glass becomes milk-white on cooling.	Insoluble. The soda goes into the coal, leaving lime behind.	Perfectly infusible; becomes grey.
4. MAGNESIA.	The carbonate is decomposed, becoming luminous and alkaline.	Like lime, but not so strongly crystalline.	Like lime.	Like lime.	After long blowing has a pale flesh-colour, only properly seen when quite cold. Phosphate and arseniate fuse and become violet-red.

EARTHS.	ALONE ON CHARCOAL AND IN THE FORCEPS.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA ON CHARCOAL.	WITH COBALT SOLUTION IN THE O. F.
5. ALUMINA.	Unaltered.	Dissolves slowly to a clear glass, becoming opaque neither by flaming nor by saturation. When much is added in fine powder, the glass is cloudy, scarcely fusible, and shows a crystalline surface on cooling.	Dissolves slowly to a clear glass, that is always clear. When much is added the undissolved part becomes semi-transparent.	Swells a little, forms an infusible compound, and the excess of soda goes into the coal.	After a strong blast assumes a fine blue colour, the intensity of which is only properly apparent on cooling.
6. GLUCINA.	Unaltered.	Dissolves largely to a clear glass, which becomes milk-white by flaming, or after cooling, when fully saturated.	As with borax.	Insoluble.	Assumes a light bluish-gray colour.
7. YTTRIA.*	Unaltered.	Like glucina.	Like glucina.	Insoluble.	0
8. ERBIA.	The yellow oxide in the R. F. becomes lighter and translucent.	Dissolves rather slowly to a clear glass, which becomes milk-white by flaming, or on cooling, when perfectly saturated.	As with borax.	Insoluble.	0
9. ZIRCONIA.	Infusible. Prepared from the sulphate it is more luminous than any other substance.	Like glucina.	Dissolves somewhat more slowly than in borax, and yields an opaque glass more quickly.	Insoluble.	Assumes a dirty, violet colour.

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* 7, 8, and 10 are the descriptions already given by Berzelius.

10. THORIA.	Unaltered.	Dissolves in small quantity to a clear glass, milk-white on cooling, if saturated; but if it appears clear on cooling it cannot be made opaque by flaming.	As with borax.	Insoluble.	0
11. SILICA.	Unaltered.	Dissolves slowly to a clear, difficultly fusible glass that cannot be made opaque by flaming.	Dissolves in very small quantity to a clear glass. The undissolved portion becomes semi-transparent.	Dissolves with lively effervescence to a clear glass.	With a little solution assumes a feeble bluish colour, becoming black or dark grey with more. The thinnest edges can be fused to a reddish-blue glass in a very hot flame.

METALLIC OXIDES AND **ACIDS**, IN ALPHABETICAL ORDER.	ALONE ON CHARCOAL, ETC.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA.	WITH COBALT SOLUTION IN THE O. F.
	IN THE OXIDIZING AND REDUCING FLAMES.				
1. TEROXIDE OF ANTIMONY, OR ANTIMONOUS ACID.	O. F. Is displaced and spreads partly over another spot. R. F. Is reduced and volatilized, coating the coal with S̈b, and tinging the flame greenish-blue.	O. F. Dissolves largely to a clear glass, yellowish while hot, colourless on cooling. On coal the dissolved acid can be driven off, so that the addition of this produces no change. R. F. The glass treated only for a short time with the O. F., becomes on charcoal grayish, and clouded from reduced Sb; but this afterward volatilizes, leaving a clear glass. With tin the glass becomes grey or black according to the degree of tion.	O. F. Dissolves with ebullition to a clear glass, only slightly yellowish while warm. R. F. The saturated glass becomes on coal, at first cloudy, afterward clear, as the Sb is reduced and volatilized. Treated with tin the glass becomes grey from reduced Sb, but on longer blowing, clear again. Tin produces a greyish cloudiness even if very little S̈b is present.	Is reduced very easily on coal in either flame, but the metal volatilizes immediately, forming a white coat of oxide of antimony.	A coat of S̈b moistened with cobalt solution and ignited in the O. F. volatilizes in part, but the remainder is more highly oxidized, and when quite cool appears dirty dark green.

METALLIC OXIDES AND ACIDS, IN ALPHABETICAL ORDER.	ALONE ON CHARCOAL, ETC.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA.	WITH COBALT SOLUTION IN THE O. F.
	IN THE OXIDIZING AND REDUCING FLAMES.				
2. ARSENOUS ACID.	Volatilizes below a red heat.	0	0	Is reduced on coal with evolution of arsenical fumes which can be recognized by their strong garlic odour.	0
3. TEROXIDE OF BISMUTH.	O. F. On platinum foil fuses easily to a dark brown mass, pale yellow when cold. On coal is reduced in O. F. and R. F. to metal, and gradually volatilized, forming a coat of yellow oxide; beyond this coat is another thinner white coat of carbonate. In the R. F. these coats disappear without colouring the flame.	O. F. Dissolves easily to a clear yellow glass, which, when little is added, is colourless on cooling. With more, the glass is yellowish-red while hot, becomes yellow in cooling, and when quite cold, is opalescent. R. F. The glass on coal is at first grey and clouded, then the oxide is reduced to metal with effervescence, and the glass becomes clear. Addition of tin accelerates the reduction.	O. F. Dissolves easily to a clear yellow glass, colourless on cooling. With much the glass can be flamed to an enamel, and with still more it becomes enamel-white of itself on cooling. R. F. On coal, especially with tin, the glass changes, becoming clear and colourless while hot, but blackish-grey and opaque on cooling.	On coal it is reduced immediately to metallic bismuth.	0
4. OXIDE OF CADMIUM.	O. F. On platinum foil is unchanged. R. F. On coal it shortly disappears and coats the surrounding coal with a reddish-brown to dark yellow powder, the colour of which is only properly seen when cold. The coal beyond the coat shows a varie-	O. F. Dissolves very largely to a clear yellowish glass, almost colourless on cooling. When strongly saturated the glass becomes milk-white by flaming, and with still more, becomes enamel-white of itself on cooling. R. F. The glass containing oxide boils on coal; the cadmium is reduced and immediately volatilized, coating the coal with dark	O. F. As with borax; the clear glass is colourless on cooling, and if saturated, appears milk-white when cold. R. F. The dissolved oxide is slowly and imperfectly reduced on coal, forming a very slight dark yellow coat, only showing its proper colour on cooling. Tin	O. F. Insoluble. R. F. On coal is immediately reduced and volatilized, coating the coal with reddish-brown to dark yellow oxide. The more remote portion of the coal assumes a variegated tarnish	0

5. SESQUIOXIDE OF CERIUM.	The protoxide is converted by the O. F. into sesquioxide Ꞓe, which remains unaltered in the R. F.	yellow to red glass, similar to that of sesquioxide of iron; on cooling, the bead is yellow. With a certain degree of saturation it can be flamed enamel-like, and with more becomes so of itself on cooling. R. F. The yellow glass becomes colourless. A strongly saturated glass becomes enamel-white and crystalline on cooling.	O. F. As with borax, but the colour disappears entirely when cold. R. F. The glass is quite colourless, both hot and cold; thus being distinguished from a sesquioxide of iron glass. It does not become opaque on cooling with any degree of saturation.	Insoluble. The soda goes into the coal while the sesquioxide is converted into protoxide, and remains behind with a light grey colour.	0
6. SESQUIOXIDE OF CHROMIUM.	Unaltered in O. F. and R. F.	O. F. Dissolves slowly, but colours strongly. With little, the hot glass appears yellow (chromic acid), but is yellowish-green on cooling; with more it is dark red when hot, but becomes yellow in cooling, and when quite cold, fine yellowish-green. R. F. The slightly saturated glass has a fine green colour, both hot and cold (Ꞓr). With more it becomes darker, or pure emerald green. Tin causes no change.	O. F. Soluble to a clear glass, reddish while hot, but dirty green when cooling, and a fine green when quite cold. R. F. As in the O. F., but the colours are somewhat darker; the same with tin.	O. F. On platinum wire dissolves to a dark brownish-yellow glass, opaque yellow on cooling. (chromic acid). R. F. The glass is opaque and green on cooling (Ꞓr). Cannot be reduced to metal on coal, but remains as green Ꞓr, while the soda goes into the coal.	0
7. PROTOXIDE OF COBALT.	O. F. Unchanged. R. F. Shrinks somewhat, and is reduced without fusing to metal, which is magnetic, and assumes a metallic lustre when rubbed in the mortar.	O. F. Colours very strongly; the glass is pure smalt-blue, both hot and cold. The strongly saturated glass is so dark blue that it appears black. R. F. As in O. F.	O. F. As with borax, but with the same quantity the colour is not quite so intense, as is seen especially on cooling. R. F. As in O. F.	O. F. On platinum wire, in very small quantity, dissolves to a transparent, feebly red mass, grey when cold. R. F. On coal is reduced to a grey magnetic powder, assuming a metallic lustre by friction.	0

METALLIC OXIDES AND ACIDS, IN ALPHABETICAL ORDER.	ALONE ON CHARCOAL, ETC.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA.	WITH COBALT SOLUTION IN THE O. F.
	IN THE OXIDIZING AND REDUCING FLAMES.				
8. OXIDE OF COPPER.	O. F. Fuses to a black globule, soon spreads out, and is reduced on the lower side. R. F. Reduced below the melting point of copper; the reduced particles show a copper lustre, but when the blast is stopped, oxidize again on the surface, becoming black or brown. Strongly heated, it fuses to a button of copper.	O. F. Colours rather strongly; a little causes a green glass while hot, and a blue when cold. With more it is dark green to opaque when hot, but greenish-blue on cooling. R. F. Saturated to a certain degree, the glass soon becomes colourless, but on cooling is red and opaque (suboxide). On coal the copper is reduced to metal, and the cold glass is quite colourless. A glass containing oxide treated on coal with tin becomes brownish-red and opaque on cooling (suboxide).	O. F. The colours are the same as with borax, but not so strong for the same amount of oxide. R. F. A rather strongly saturated glass becomes dark green, and on cooling changes at the moment of solidifying to an opaque brownish-red (suboxide). A glass containing but little, if treated on coal with tin, appears colourless while hot, but becomes brownish-red and opaque when cold.	O. F. Dissolves on platinum wire to a clear green glass, losing colour and becoming opaque on cooling. R. F. On coal is reduced very easily to metal, which can be fused to one or several buttons by a sufficient heat.	0
9. SESQUIOXIDE OF DIDYMIUM.	O. F. Infusible. R. F. At a high temperature loses its brown colour and becomes grey. (*Berzelius.*)	O. F. Soluble to a clear rose-red glass, unaltered in the R. F. (*Hermann.*)	Dissolves with more difficulty than in borax, but when strongly saturated is distinctly rose-red. (*Hermann.*)	Insoluble. The soda goes into the coal, and the grey oxide remains behind. (*Berzelius.*)	0
10. TEROXIDE OF GOLD.	Is reduced on ignition in either flame, and can be easily melted to a button.	O. F. Reduced without dissolving; on coal can be fused to a button. R. F. The same.	As with borax.	As with borax; the soda goes into the coal.	0

11. **OXIDE OF INDIUM.**	ed, but on cooling is lighter again, and does not fuse. R. F. Gradually reduced and volatilized, depositing a coat on the coal. A very distint violet-coloured flame is produced.	while hot, colourless on cooling, and cloudy when much is added. R. F. The glass is unchanged. On coal the oxide is reduced, volatilizes, and coats the coal again with oxide. The violet flame is perceptible notwithstanding the soda.	As with borax, but the glass when treated with tin on charcoal, becomes grey and cloudy on cooling.	R. F. Is reduced on coal, and the metal partly volatilizes, coating the coal with oxide, while it may partly be observed as almost silver-white globules in the fused salt.	0
12. **BINOXIDE OF IRIDIUM.**	Heated to ignition is reduced, but cannot be fused.	O. F. Reduced without dissolving, but cannot be united to a button, even on coal. R. F. The same.	As with borax.	As with borax; the soda goes into the coal.	0
13. **SESQUIOXIDE OF IRON.**	O. F. Unchanged. R. F. Becomes black and magnetic (protosesquioxide).	O. F. With little the glass is yellow when hot, colourless on cooling; with more it is red while hot, yellow when cold; with still more dark red while hot and dark yellow on cooling. R. F. The glass becomes bottle-green (Fe F̶e). On coal with tin it becomes at first bottle-green, but on longer blowing, vitriol-green (protoxide).	O. F. With a certain amount the hot glass is yellowish-red, but becomes first yellow while cooling, then greenish, and finally colourless. With very much it is dark red when hot, and in cooling becomes brownish-red, dirty green, and finally a less brownish-red. The colours disappear on cooling much sooner than with borax. R. F. The glass seems unchanged with a small amount of oxide; with more it is red while hot, and on cooling becomes, first yellow, then greenish, and finally reddish. With tin on coal it becomes green in cooling, and finally colourless (protoxide).	O. F. Insoluble. R. F. Is reduced on coal, yielding a grey, magnetic metallic powder when the particles of coal are washed away.	0

METALLIC OXIDES AND **ACIDS,** IN ALPHABETICAL ORDER.	ALONE ON CHARCOAL, ETC.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA.	WITH COBALT SOLUTION IN THE O. F.
	IN THE OXIDIZING AND REDUCING FLAMES.				
14. OXIDE OF LANTHANUM.	Unchanged.	O. F. Dissolves to a clear, colourless glass, that becomes enamel-white by flaming when saturated to a certain extent.and when strongly saturated becomes enamel-like of itself on cooling. (*Berzelius.*) R. F. As in O. F.	As with borax.	Insoluble. The soda goes into the coal, leaving the grey oxide behind. (*Berzelius.*)	0
15. OXIDE OF LEAD.	Minium heated on platinum foil blackens, and heated to incipient redness, changes to yellow oxide. At a higher temperature this oxide fuses to a yellow glass. On coal is immediately reduced by either flame with effervescence, and the metal gradually volatilizes, coating the coal with yellow oxide; behind this is another thinner coat of white carbonate. The coats disappear under the R. F., ting-	O. F. Dissolves easily to a clear yellow glass, colourless on cooling, and which, with a larger quantity becomes opaque by flaming; with still more, it becomes opaque and enamel-yellow of itself on cooling. R. F. The glass spreads out on coal and becomes cloudy; on continuing the blast, the oxide reduces with effervescence to metal, and the glass becomes clear again.	O. F. As with borax, but requiring more of the oxide to obtain a glass yellow when hot. R. F. The glass becomes greyish and cloudy on coal. With more oxide, a yellow coat is formed on the coal. With tin, the glass becomes cloudier and darker grey, but never quite opaque.	O. F. On platinum wire dissolves easily to a clear glass, becoming yellowish and opaque on cooling. R. F. Is immediately reduced on coal to metal, which afterward coats the coal with oxide.	0

16. SESQUI-OXIDE OF MANGANESE.	O. F. Infusible. In a hot enough flame both the sesqui-and peroxide are altered to proto-sesquioxide, yielding oxygen, and turning brownish-red. R. F. The same.	O.F. Colours very intensely. The hot glass is violet (amethyst), but on cooling becomes violet-red. An excess renders the glass quite black and opaque, the colouring being only visible by pinching the still soft glass with the forceps. R. F. The glass becomes colourless (protoxide). With a very dark glass this succeeds better on coal and with tin.	dition is requisite to colour the glass, which then has a brownish-violet colour while hot, and is reddish-violet on cooling, but never opaque. When the glass contains so little as to be colourless, the colour can be produced by nitre (*vide* manganese. A glass containing oxide bubbles and yields gas at a high temperature. R. F. The glass very soon becomes colourless, and remains then perfectly quiet.	O. F. On platinum wire or foil, in very trifling quantity, dissolves to a clear, transparent green mass, becoming opaque and bluish-green on cooling (manganate of soda). R. F. Not reducible to metal on coal; the soda sinks in, leaving the protoxide behind.	0
17. PROTOXIDE OF MERCURY.	Is instantly reduced and volatilized.	0	0	Heated to redness in the matrass it is reduced and vaporized (also when heated alone). The vapours condense in the neck to a metallic coat, which can be united to globules by carefully tapping on the matrass.	0
18. MOLYBDIC ACID.	O.F. Fuses, spreads out, volatilizes, and forms at a certain distance, a yellowish, pulverulent coat, consisting of small crystals nearest the assay. The coat becomes white on cooling, and the	O. F. Dissolves easily and largely to a clear glass, yellow while hot, and colourless on cooling. A very large addition produces a glass dark yellow to dark red while hot, and opaline to a bluish-grey enamel when cold.	O. F. Dissolves easily to a clear glass, which is yellowish-green while hot, if a moderate amount is added, but becomes nearly colourless on cooling. On coal, becomes quite dark, and on cooling, is fine green, owing to $\ddot{\mathrm{Mo}}$ by the carbonic oxide formed.	O. F. On platinum wire fuses with effervescence to a clear glass, milk-white *on cooling*. R. F. On coal fuses with effervescence at first, but afterward the fused mass is absorbed by	0

METALLIC OXIDES AND ACIDS, IN ALPHABETICAL ORDER.	ALONE ON CHARCOAL, ETC.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA.	WITH COBALT SOLUTION IN THE O. F.
	IN THE OXIDIZING AND REDUCING FLAMES.				
MOLYBDIC ACID. (Continued.)	crystals colourless. Beyond this coat is a thinner, non-volatile film of binoxide (M̈o), which on cooling is dark copper-red, and has a metallic lustre, p. 67. R. F. The greater part sinks into the coal, and can be reduced by a good flame to metal, which is obtained as a grey powder after washing away the particles of coal.	R. F. The glass produced in the O. F. becomes brown with a certain degree of saturation, and with still more, is opaque (M̈o). In a good flame black flocks of M̈o separate, and can be very distinctly seen in the yellowish glass when it is pinched out flat.	R. F. The glass formed in the O. F. becomes quite dark dirty-green, but purer green on cooling. The same on coal. With tin, a somewhat darker green (protoxide?).	the coal, and the greater part of the acid is reduced to metallic molybdenum, which can be obtained as a steel-grey powder by washing away the coal.	0
19. PROTOXIDE OF NICKEL.	O. F. Unchanged. R. F. On coal is reduced to a metallic, coherent, and infusible powder, which, by friction in the mortar, assumes a metallic lustre, and is decidedly magnetic.	O. F. Colours quite intensely; a little colours the hot glass violet, but when cold, a pale reddish-brown; with more the colours are darker. R. F. The glass becomes grey and cloudy, or quite opaque, owing to finely divided metallic nickel. On continuing the blast the reduced metallic particles collect together without fusing and the glass becomes colourless. On coal, especially with tin, the reduction is more rapid, and the nickel	O. F. Dissolves to a reddish-glass, yellow on cooling. With a large addition the hot glass is brownish-red, and becomes reddish-yellow when cold. R. F. On platinum wire unchanged. On coal, with tin, becomes at first opaque and grey, but after longer blowing all nickel is reduced and the glass becomes colourless.	O. F. Insoluble. R. F. On coal is easily reduced to white metallic particles, which, after the coaly matters are washed away, follow the magnet readily.	0

20. NIOBIC ACID.*	O. F. Becomes yellowish, but is white again on cooling. R. F. The same.	clear colourless glass, becoming opaque by flaming with a certain addition, and with more becomes opaque of itself when cool. R. F. A glass which, after treatment in the O. F. becomes opaque of itself, on cooling remaining unaltered.	a clear glass, yellow while hot, but colourless on cooling. R. F. With a very large addition the glass becomes brown. The addition of sulphate of iron causes a blood-red bead.	fuses with effervescence; with more soda goes into the coal. R. F. The same. It cannot be reduced to metal.	Assumes a light grey colour.
21. HYPONIOBIC ACID.	Like niobic acid.	O. F. Dissolves easily to a clear glass, which, with the addition of a very large amount, can be made opaque by flaming. R. F. A glass formed in the O. F., which opalesces on cooling, becomes clear again. With more, it becomes cloudy, and bluish-grey on cooling; with a very large amount it becomes quite opaque and bluish-grey.	The acid prepared from the sesquichloride (*unterniobchlorid*) behaves as follows: O. F. Added in large quantity it dissolves to a greenish-blue glass, which, on continued blowing becomes clear and colourless. R. F. Becomes pure blue (on coal pale brown), and with addition of sulphate of iron becomes blood-red. The acid prepared directly from columbite, and purified, yields only a brown glass in the inner flame.	Like molybdic acid.	While hot is grey, but on cooling becomes dirty-green. With too strong a heat it sinters, and the perfectly cold assay appears dark grey where most strongly heated.
22. BINOXIDE OF OSMIUM.	O. F. Changed into osmic acid (Os), which volatilizes in penetrating and suffocating vapour, having a very irritating effect on the eyes. R. F. Reduced to a dark brown infusible metallic powder, which can be very easily oxidized to osmic acid.	0	0	Is very easily reduced to an infusible metallic powder, which can be obtained pure by washing away the coal, etc.	0

* Vide note under "Exam. for Tantalum and Niobium."

METALLIC OXIDES AND **ACIDS** IN ALPHABETICAL ORDER.	ALONE ON CHARCOAL, ETC.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA.	WITH COBALT SOLUTION IN THE O. F.
	IN THE OXIDIZING AND REDUCING FLAMES.				
23. PROTOXIDE OF PALLADIUM.	Is reduced at a red heat, but the metallic particles are infusible.	O. F. Reduced without dissolving, but the metallic particles cannot be united to a button even on coal. R. F. The same.	As with borax.	Insoluble. The soda sinks into the coal, leaving the palladium as an infusible powder.	0
24. BINOXIDE OF PLATINUM.	Like palladium.	Like palladium.	Like palladium.	Like palladium.	0
25. SESQUIOXIDE OF RHODIUM.					
26. SESQUIOXIDE OF RUTHENIUM.					
27. OXIDE OF SILVER.	Reduced easily to metallic silver, which fuses to one or more globules.	O. F. Partly dissolved and partly reduced. The cold glass is opalescent or milk-white, according to the amount dissolved. Metallic silver fused with borax in a clay capsule yields the same glass. R. F. On coal the glass is at first greyish, from reduced silver, but after all the silver is separated and fused to a button, the glass becomes clear and colourless.	O. F. Both the oxide and the metal produce a yellowish glass. A glass containing much oxide is opalescent on cooling, and when held against the daylight appears yellowish, against the lamp-flame, reddish. R. F. As with borax.	Is instantly reduced to one or more globules of silver, while the soda sinks into the coal.	0

28. TANTALIC ACID.	O. F. Becomes slightly yellow, but is white again when cold. R. F. The same.	O. F. Dissolves easily to a clear glass, which, with a certain amount appears yellowish while hot, colourless on cooling, and can be made opaque by flaming. With still more the glass becomes enamel-white of itself on cooling. R. F. As in O. F.	O. F. Dissolves largely to a clear glass, which, with a very large amount, is yellowish while hot, but colourless on cooling. R. F. The above glass undergoes no alteration.	what more than an equal volume of soda fuses on coal to a bead with effervescence, and soon spreads out; with more soda sinks into the coal. R. F. The same. It cannot be reduced to metal.	tion appears light grey, but on cooling assumes a feeble red colour, quite similar to that of magnesia. If not entirely free from alkalies, it sinters and becomes bluish-black.
29. TELLUROUS ACID.	O. F. Fuses, and is reduced with effervescence; the reduced metal volatilizes immediately, forming a white coat of T̈e, which usually has a red or dark-yellow border. R. F. The same. The outer flame is also tinged with bluish-green.	O. F. Dissolves to a clear colourless glass, which on coal becomes grey, from reduced metal. R. F. A glass obtained on platinum wire in the O. F. becomes first grey, and after all the tellurium is reduced and volatilized, it becomes colourless again. A coat of T̈e is also formed.	As with borax.	Dissolves on platinum wire to a colourless glass, white on cooling. On coal is reduced and volatilized, forming a coat of Te.	0
30. BINOXIDE OF TIN.	O. F. The protoxide burns like tinder and is changed to S̈n. The binoxide becomes strongly luminous, and is yellowish while hot, but dirty yellowish-white when cold. R. F. Is reduced to metal by a continued and strong flame, a trifling coat of S̈n being generally formed near the assay.	O. F. Dissolves in trifling quantity, and very slowly, to a clear colourless glass, remaining clear on cooling, and not becoming opaque by flaming. A saturated and perfectly cold glass, when heated to low redness becomes opaque, loses its spherical shape, and shows indistinct crystallization. R. F. A not saturated glass is unchanged. On coal some tin can be reduced from a glass containing much oxide	O. F. Dissolves in trifling quantity, and very slowly, to a colourless glass, clear both hot and cold. R. F. This glass is unchanged on coal and on platinum wire.	O. F. On platinum wire combines with the soda with effervescence to a swollen, infusible mass. R. F. Reduced to tin on coal.	Assumes a bluish-green colour, not properly seen until perfectly cold.

METALLIC OXIDES AND **ACIDS,** IN ALPHABETICAL ORDER.	ALONE ON CHARCOAL, ETC.	WITH BORAX ON PLATINUM WIRE.	WITH SALT OF PHOSPHORUS ON PLATINUM WIRE.	WITH SODA.	WITH COBALT SOLUTION IN THE O. F.
	IN THE OXIDIZING AND REDUCING FLAMES.				
31. TITANIC ACID.	O. F. Assumes a yellow colour, but is white again on cooling. No further change. R. F. The same.	O. F. Dissolves easily to a clear glass, which, if much is present, is yellow while hot, but colourless on cooling, and becomes opaque by flaming; with very much it becomes enamel-white of itself on cooling. R. F. A small addition yields a yellow glass; more yields a dark yellow to brown glass (sesquioxide). A saturated glass becomes enamel-blue by flaming.	O. F. Dissolves easily to a clear glass, yellow while hot, if much is present, but colourless on cooling. R. F. This glass remains yellow while hot, but reddens on cooling, and assumes a fine violet colour ($\ddot{Ti}$). If the acid contains iron, the glass becomes brownish-yellow to brownish-red on cooling. On coal with tin it becomes violet, unless too much iron is present.	**O.F. Dissolves with effervescence on coal to a dark yellow glass, which crystallizes on cooling, and thereby evolves so much heat that the globule glows strongly again. When perfectly cold the glass is greyish-white to white. R. F. The same. It cannot be reduced to metal.**	**Assumes a yellowish-green colour, similar to oxide of zinc, but not so fine.**
32. TUNGSTIC ACID.	O. F. Unchanged, unless the temperature is so high that it is reduced to binoxide (W), owing to the formation of carbonic oxide gas. R.F. Becomes black (binoxide), but is infusible.	O. F. Dissolves easily to a clear colourless glass. With a rather large addition it appears yellow while hot, and with more can be made enamel-like by flaming; with still more it becomes enamel-white of itself on cooling. R. F. With a small quantity the glass is unchanged; with more it becomes yellow to dark yellow, and yellowish-brown on cooling ($\dot{W}$). On coal a smaller amount produces this reaction, and with tin darker colours are produced, when not too strongly saturated.	O. F. Dissolves easily to a clear colourless glass, only yellow when strongly saturated and hot. R. F. The above glass, after short treatment, is dirty-green, but becomes blue on cooling ($\dot{W}$, $\dot{W}$); after too long a blast it is bluish-green when cold. On coal, especially with tin, it becomes dark green ($\dot{W}$). If it contains iron, the hot glass on the wire appears yellow, but becomes brownish-red (blood-red) on cooling, as with Ti containing iron. With tin on coal the glass becomes blue, if the amount of iron is inconsiderable.	**O. F. On platinum wire dissolves to a clear dark yellow glass, becoming crystalline and opaque-white or yellowish on cooling. R. F. With a little soda on coal a large amount may be reduced to metallic tungsten; with more soda the whole sinks into the coal, and a yellow compound of oxide of tungsten and soda, having a metallic lustre, is formed.**	0

33. **Sesquioxide of Uranium.**	O. F. Infusible, but assumes a dirty, dark yellowish-green colour (U U). R. F. Becomes black and shows this also on friction in the mortar (protoxide).	O. F. Like sesquioxide of iron, but with an equal amount the colours are lighter. The strongly saturated glass can be made enamel-yellow by flaming. R. F. Like Fe. The green (U U) glass, saturated to a certain extent, can be made black by flaming, but becomes neither enamel-like nor crystalline. With tin on coal becomes dark green $(\dot{U})$.	O. F. Dissolves to a clear, yellow glass, yellowish-green on cooling. R. F. The above glass becomes dirty green, but on cooling, pure and fine green (U U). With tin on coal the green becomes darker (U).	O. F. Insoluble. With a slight addition of soda shows signs of fusing; with more the mass becomes yellowish-brown; with still more, the oxide goes into the coal. R. F. The same; it cannot be reduced to metal.	0
34. **Vanadic Acid.**	Fusible. The portion in contact with the coal is reduced and goes into the coal; the remainder assumes the colour and lustre of graphite, and is protoxide of vanadium $(\dot{V})$	O. F. A little dissolves to a clear colourless glass, but more gives a yellow glass, greenish-yellow on cooling. R. F. The above glass changes, appearing brownish while hot, and fine chrome-green on cooling $(\ddot{V})$.	O. F. Dissolves to a clear glass; if not in too small quantity, dark yellow while hot, and light yellow on cooling. R. F. As with borax.	Fuses together with it, and goes into the coal.	0
35. **Oxide of Zinc.**	O. F. Becomes transiently yellow, and is infusible, but strongly luminous when ignited. R. F. Gradually disappears, being reduced, volatilized and again oxidized. Part of the oxide forms a new, distinct coat on another part of the coal, being yellowish while hot, and white on cooling.	O. F. Dissolves easily and largely to a clear glass, yellowish while hot, but colourless on cooling; with more this becomes enamel-like by flaming, and with still more, becomes so of itself on cooling. R. F. The saturated glass becomes cloudy and greyish by the first blast (since some oxide is separated by the imperfect fusion), but after longer blowing is clear again. On coal is gradually reduced, volatilized and reoxidized, forming a coat on the coal.	As with borax.	O. F. Insoluble. R. F. On coal is reduced, volatilized, and reoxidized, giving a zinc coat. With a good flame the zinc flame may even be produced.	Assumes a fine, yellowish-green colour, most distinct when perfectly cold.

B. General Rules for Qualitative Blowpipe Examinations, by which the separate Constituents of Compound Substances can be detected with the partial Aid of the Wet Process.

In the examination of compound substances with the aid of the blowpipe the wet process is frequently indispensable when all of the ingredients are to be detected; but even then the blowpipe is advantageously used, not only in carrying out various necessary operations, but also as a means of controlling the results and further examining the isolated constituents.

Before proceeding to decompose the substance by the wet way, its behaviour before the blowpipe should be ascertained, and from this the nature of the compound inferred; whether it is a salt of the alkalies, earths, or heavy metals, or a silicate, and whether the compounds contain easily reducible metallic oxides; further, whether it is a combination of metallic oxides, or of sulphides or selenides, or of various metals with one another, in which latter division the metallic arsenides and tellurides are also to be placed. The further examination is essentially facilitated by knowing under what class of compounds the unknown substance belongs.

When the compounds are insoluble in water, the most usual solvent is hydrochloric acid, and if solution does not take place at the usual temperature, the glass is heated over the spirit-lamp. Effervescence indicates either the presence of a carbonate, when the carbonic acid gas escapes without any odour; or of a metallic oxide at a high state of oxidation, and then chlorine gas escapes and is recognized by its pungent odour. The latter phenomenon occurs, for example, when the mineral contains sesquioxide or binoxide of manganese, which are transformed into protochloride. The solution is then diluted with distilled water and examined for the various earths and acids, as will be directed under the separate examinations.

The testing of silicates with hydrochloric acid is of especial importance. Quite a number are wholly decomposed by it, a gelatinous or pulverulent (and then usually rather voluminous) state.*

When the silicate is entirely decomposable, the whole is diluted with water, filtered, and the filtrate examined, as will be directed under the examination of silicates, for the various earths. Should it not be wholly decomposed, another small portion must be decomposed by fusion with carbonate of soda and borax, as will be described directly.

In certain cases it is necessary to fuse a substance with nitre when examining it for a single ingredient, which is thus more highly oxidized and combined as an acid with the potassa of the nitre, from which it can be more readily separated and then recognized. Sometimes, also, it is necessary to fuse a substance with bisulphate of potassa, and to dissolve the fused mass in water, in order either to free it at once from certain constituents, or to convert the whole into sulphates, and then proceed with the separation of the different constituents after solution in water.

Decomposition of the substances by fusion with soda and borax, and treatment of the fused mass with hydrochloric acid.

Seventy-five to one hundred milligr. of the very finely powdered substance being mingled with soda and borax † in the agate mortar, the whole is wrapped in a soda-paper cylinder, made of fine filter-paper, and fused before the blowpipe in a cylindrical hole on charcoal, or in a charcoal crucible. The quantity of flux depends on the fusibility of the substance. Generally, one part by weight of soda and one of borax suffices; but the borax must be increased up to two parts when the substance contains much magnesia, alumina, glucina, or zirconia, while a considerable amount of barite requires an increase of both borax and soda.

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* To determine whether silicates and other combinations of the earths and metallic oxides are decomposed by acids, the fine powder is boiled for some time with the acid, and a portion of the fluid tested with ammonia and phosphate of soda. If a considerable precipitate is formed, decomposition has taken place, but if only a few flocks are thrown down, the substance is either not decomposed, or only with difficulty.

† The boracic acid being combined with the soda, exercises no injurious effect during the subsequent decomposition of the fused compound by the wet

The O. F. may be employed if the substance has been found free from easily reducible oxides, but otherwise, as in the case of certain slags, the R. F. must be used, so as to reduce and separate these oxides in the metallic state.

Usually the quantity of reducible oxides is so small that *they cannot easily be reduced to a single button*, and then about *sixty to eighty milligr. of silver, or, still better, gold,* should be added in the form of a button and the charge treated with the R. F., just as directed for the charge in the quantitative copper assay. During this operation the earthy constituents and the difficultly reducible oxides dissolve in the glass which is formed by the borax and soda, and melt to an easily fusible bead. The acids of arsenic and the easily reducible oxides are reduced, while, in presence of sulphuric acid, there is a partial formation of sulphide of sodium, and part of the sulphur combines with the reduced metals. The reduced arsenic is partly taken up by these metals and partly volatilized, while the non-volatile reduced metals unite together and melt with the silver or gold to an easily fusible globule, which goes to one side of the glass. The oxides of the metals that remain dissolved in the glass are present in the lowest stage of oxidation.

Such a fusion, whether with the O. F. or R. F., must be accomplished with an active flame and with proper patience, since otherwise no thorough decomposition of the substance can be effected. The fused glass must be quite fluid, as clear as possible, and free from bubbles and metallic particles. If the glass continues to froth or show bubbles after long blowing, this is a sign that either the solution of the non-reducible portions, or the reduction of the reducible oxides, is not completed, and the fusion must be continued with a lively flame.

An assay which has been fused in the O. F. is taken from the coal when it has solidified, and after cleaning it from any adherent coal with the knife and brush, is first broken up in the steel mortar, or between paper on the anvil, and then pulverized in the agate mortar. It must be entirely reduced to powder, otherwise portions of the glass are apt to remain must be pulverized immediately, or else the fused glass will absorb moisture readily from the air, becoming tough and difficult to pulverize.

When the assay has been fused in the R. F., and a metallic button reduced from it, or when it is presumed that the silver or gold added has been melted to a globule with the reduced metals, the assay must still be kept in a quite fluid state by means of a good R. F., and made to flow slowly from one point of the coal to another, until it is certain that the glass is quite free from globules of metal and bubbles, while the metal is united to one globule beside it. After satisfactorily accomplishing this the blast is stopped, and the assay set aside until quite cold, when it is removed with the forceps, the metallic button separated from the glass in the steel mortar, or between paper on the anvil, and the glass pulverized after cleansing it from any adherent coal. When the R. F. has not been pure or strong enough, part of the reducible oxides may remain and exert an injurious influence upon the further decomposition of the fused glass.

The oxides which can be easily reduced by the foregoing treatment, and thus separated from the earths and non-reducible oxides are: the acids of arsenic and antimony, and the oxides of silver, mercury, copper, bismuth, thallium, lead, tin, zinc, indium, cadmium, and nickel. Tellurium, osmium, gold, platinum, iridium, rhodium, and palladium only occur in nature in the metallic state, and can therefore readily be separated from the non-reducible oxides and earths by the addition of silver or gold. The volatile metals either escape entirely in fumes during the fusion, or only in part, some of them coating the coal, while those that remain unite with the added silver or gold. The manner of conducting the further examination of the reduced metals may be inferred from the remarks upon alloys in the detailed examinations which follow.

The oxides of the heavy metals which cannot be reduced by fusion in the R. F. with soda and borax, are: the oxides of chromium, uranium, cobalt (in the absence of arsenic acid, or when the cobalt is not present in too great quantity), iron, manganese, and cerium; and the following acids: molybdic, tungstic, tantalic, niobic, and titanic. The above oxides can, however, be readily separated again from the earths, for the most part, and recognized before the blowpipe, as directed in the various qualitative examinations.

After finely pulverizing the fused glass, it is moistened with an abundance of water in a porcelain dish, and then as much hydrochloric acid is added as will dissolve all of the powder, and leave some free acid. The dish is placed upon the evaporating ring, over the lamp flame, and the powder stirred with a slender glass rod, until the soluble portions have been separated from the insoluble. The presence of sulphide of sodium, always formed if the substance contains sulphuric acid or sulphur, causes evolution of sulphuretted hydrogen; the other constituents, excepting silica, are converted into chlorides and dissolved by the dilute acids. Occasionally almost all of the silica also goes into solution. Since usually only combinations of silicic acid are decomposed by fusion with soda and borax, it rarely happens that molybdic, tungstic, tantalic, niobic, and titanic acids are here met with. After effecting the solution, the whole is evaporated to dryness, and this should be done where the vapours may not escape into the laboratory, in case too much free acid is present. The evaporation should be conducted gradually, especially toward the end, and carried to dryness, so as to remove the excess of acid, and to render compact the silica, which separates in a gelatinous state during the process.

When the solution has been evaporated to dryness, so that only the slightest possible odour of escaping acid vapour can be perceived, the mass is moistened with hydrochloric acid, and after some time is covered with distilled water. Then the dish is set over the lamp flame, in order to dissolve the chlorides and separate them from the insoluble portion, which in substances decomposed in this way generally consists of silica alone. The silica can be very readily separated from the solution by filtration and washing with water, after which it may, if necessary, be tested B. B. with soda.

Sesquioxide of iron is reduced during the fusion to protoxide, and is not perfectly restored to the state of sesquioxide during the treatment with hydrochloric acid, and least perfectly when there is very much iron present. This being, however, essential to the certain detection of the separate ingredients, the filtrate from the silica, with the first portions of the wash water, must be boiled in a test-tube, with a few drops of nitric acid, so as to convert the protoxide of iron into sesquioxide. The nitric acid necessary for the peroxidation of the iron may be added at once, before the evaporation, when it is not desirable to have regard to the formation of sulphuretted hydrogen, which may ensue on treating the fused assay with hydrochloric acid.

The bases contained in the filtrate from the silica are separated by methods that will be given under the detailed qualitative examinations for different earths.

Fusion of substances with nitre or bisulphate of potassa.

This fusion is sometimes performed simply in the loop of a platinum wire, but oftener in the platinum spoon.* Powdered and friable substances are at once mixed in a finely pulverized state with the necessary amount of nitre in the agate mortar; metallic compounds and alloys which cannot be powdered should be divided as finely as possible by hammering or filing. The quantity of nitre depends upon the character of the substance to be oxidized; ordinarily three to four volumes are employed, unless the substance has a very high specific gravity. When but one ingredient is sought for, the fusion may be performed on platinum wire, after mixing the whole into a paste with water. A portion is smeared in the loop and fused in the O. F. until it ceases to foam, when a fresh portion is smeared on and fused, continuing the operation until the volume of the fused mass is so great that it would no longer adhere to the wire. The wire should be kept in an inclined position, with the loop turned downward, as the nitre has a tendency to flow down the wire.

When it is suspected that the substance contains an extremely small amount of the body sought for, or when it is designed to oxidize several ingredients, in order to treat them further in that state, or if an alloy which cannot be powdered is under treatment, a somewhat larger quantity must be used, and the fusion performed in the platinum spoon. The whole mixture should not, however, be poured into the spoon at once, but at first only small portions, since gases and vapours escape during the fusion, and may easily cause the melting mass to run over. At first the bottom of the spoon is heated with the O. F., which is then directed into the spoon, and the whole fused until it becomes quiet. The remainder of the mixture is then added in similar portions, and fused after each addition until it ceases to froth strongly. The position of the spoon should meanwhile be altered, so that every part of the mixture may be reached by the flame, and the spoon always appear red-hot.

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* The nitre oxidizes the surface of the platinum, it is true, but so slightly that its effects on the spoon need not be feared.

Only those alloys can be fused with nitre in the spoon which oxidize readily, and do not combine with the platinum at the temperature which can be produced by the blowpipe. The fusion of such compounds with nitre is properly limited to the detection of very trifling quantities of arsenic in metals from which it can only be separated with difficulty, and which are very infusible, *e.g.*, nickel and cobalt.

The fusion of substances with bisulphate of potassa is always effected in the larger platinum spoon, in the same way as with nitre, and most suitably over the spirit-lamp. The substance must be perfectly dried, previously reduced to fine powder, and elutriated, if decomposed with difficulty. If melted by the blowpipe flame, the too strong heat is very liable to partially expel the sulphuric acid from some of the sulphates formed by the fusion; but if the spirit flame is employed, and the spoon at first only kept directly over the tip of the flame, until most of the gases have escaped, and then held deeper in it, the heat acts equally upon the bottom of the spoon all around, the fusing mass is only brought to low redness, and the resulting salts are not destroyed.*

When much of the bisulphate must be used, the spoon frequently becomes filled before all of the mixture has been charged, and then the fluid mass must be poured out upon the anvil, and the remainder of the mixture fused. It is, indeed, always advisable to pour out the mass, as it can then be readily pulverized in the steel mortar when cold, and thus more quickly dissolved in water. The quantity of bisulphate of potassa depends upon the various constituents of the substance to be fused; thus, for protoxide of iron 3·6 parts by weight of bisulphate are required to convert it into sulphate; for lime, 4·5; for magnesia, 6; for alumina, 7·8. It is always more prudent to employ rather more of the acid salt than is exactly required, since alumina and the oxides of iron are liable to lose a part of their combined sulphuric acid at a long-continued high temperature.

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* It is advantageous in these fusions to surround the flame with a sheet-iron cylinder, reaching about to the top of the flame, and which rests on the glass lamp, having several openings at its lower end.

The firm mass resulting from the fusion of a substance with nitre or bisulphate of potassa, whether on platinum wire or in the spoon, when not poured out cannot well be pulverized and thus dissolved in water, because the platinum may easily be injured in detaching it. It is therefore necessary to lay the wire or spoon with the fused mass in a porcelain dish of suitable capacity, pour over it the amount of water necessary for solution, and set the vessel upon the evaporating ring over the lamp. As the water becomes hot the mass generally separates from the platinum and can be crushed with the pestle of the agate mortar. In most cases the water may be heated to boiling and the salt thus readily dissolved; but when a substance containing titanic acid has been fused with bisulphate of potassa, in order to make the titanic acid soluble, and the fused mass has been covered with more water than was exactly required for its solution, the latter must not be boiled, or else the titanic acid will be imperfectly dissolved, and that which had been dissolved at a lower temperature will be thrown down again.

These operations in the wet way afford residues and precipitates, which after filtration are to be further examined, and must therefore generally be dried. When there is enough of the mass the filter is spread open upon a double layer of blotting-paper, the mass scraped off with a spatula, transferred to a porcelain dish, and dried over the lamp. When there is but little of it, the folded filter is held up to the light, the empty portion cut away with the scissors, and the remainder, with the adhering mass, dried immediately in the dish over the lamp. The dry paper is then doubled together, hung upon a platinum wire, ignited at one end, and burned over a clean porcelain dish, which receives the residue, mingled with a little coal. These coaly particles may be very easily burned away in the platinum spoon, but this is not necessary when the dry mass is to be further treated with fluxes, as they are thereby consumed.

The chemical operations ordinarily performed in making examinations in the wet way, such as precipitating, decanting, filtering, washing, etc., need not be further mentioned here, since probably every one who engages in blowpipe analysis will have some knowledge of them, or the requisite information can be obtained from any manual of chemical analysis.

II. QUALITATIVE EXAMINATION OF MINERALS, ORES, AND METALLURGICAL PRODUCTS BEFORE THE BLOWPIPE FOR METALLIC AND NON-METALLIC ELEMENTS.

A. Examinations for Alkalies and Earths.

1. Potassa, K.

Its occurrence in the mineral kingdom.

Potassa is never found free, but only in combination with chlorine and sulphuric and nitric acids in certain salts, and with silicic acid in several silicates.

Examination for Potassa.

In the easily fusible potassa salts, excepting phosphate and borate, and in the compounds of potassium with chlorine, bromine, etc., this alkali is at once recognized by fusing a small portion on a loop of platinum wire with the tip of the blue flame. In the complete absence of soda, and with a clean wire, the outer flame is coloured more or less strongly violet.

Soda renders the flame more or less yellow, and lithia red, so that in their presence this simple test will not show the presence of potassa at all, or not with perfect certainty. Sometimes when the amount of soda or lithia is very trifling, the outer flame has so distinct a violet colour near the assay, that the reaction may be satisfactory; but if the soda amounts to a few per cent. even, this coloration becomes imperceptible. In this case the method proposed by Harkort may be adopted. Some borax, to which a little boracic acid has been added, is fused to a bead on platinum wire, and in this enough pure protoxide of nickel, free from cobalt, is dissolved to render the glass brownish when cold. Not too little of the salt to be examined for potassa is then added to this glass and fused with it in O. F., after which the cold glass is examined. If it has assumed a bluish colour, potassa is an essential constituent of the salt. This test, applicable only when the salt contains a very large amount of potassa, depends upon the fact observed by Lampadius, that protoxide of nickel colours the potassa glass blue, and the soda, on the contrary, brown.

The simplest means of detecting potassa with certainty in salt, in which, owing to a greater or less amount of soda, the violet coloration of the flame cannot be recognized, consists, according to Cartmell, in viewing the colour of the flame through *deep blue* cobalt glass, or a stratum of indigo solution. The presence of potassa is recognized, according to the thickness of the intervening medium, by the violet or poppy-red (*ponceau-rothe*) colour, while a very large amount of soda produces a blue colour, and a smaller quantity is not perceptible.

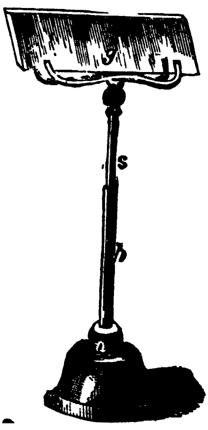

Fig. 63.

The flame of a Bunsen burner, or the blue blowpipe flame, may be employed. In the latter case a small stand renders the observation more convenient. In the small tube, *h*, fastened in an iron or lead foot, the wire, *s*, is made to slide at pleasure, and it supports in a clamp the cobalt glass, *g*. A few experiments will determine the proper situation for the glass between the eye and the flame, and give practice in holding the platinum wire with the assay in the tip of the blue flame, which is not visible through the glass. For the indigo solution a small open vessel may be formed by means of glass strips and a suitable cement, and then also set upon a stand.

If the assay contains substances which produce a luminous flame, as in the separation of carbon from burning organic matter, these must first be removed by igniting the assay, since otherwise the same violet colour will be perceived through the glass as that caused by potassa. The violet and red rays proceeding from the glowing wire must likewise not be confounded with the proper potassa coloration, which rather extends out from the assay toward the tip of the flame.

Viewed through moderately thick cobalt glass, or a thin stratum of indigo solution, the lithia flame is carmine-red, but through very dark or thick glass, or a thicker stratum of solution, it ceases to be visible, while the red potassa flame is still distinctly seen, and therefore when potassa is to be detected in presence of lithia, a thicker or darker glass must be used, the effect of which has been previously tested with pure salts.

According to observations by Merz, the lithia flame is invisible through green glass, while the potassa and baryta flames appear bluish-green, and that of soda orange-yellow. In the case just mentioned, the thicker cobalt glass or indigo solution may therefore be replaced by green glass.

The potassa in silicates cannot always be detected with certainty by the coloration of the flame, because these compounds nearly always contain more or less soda, which prevents the reaction; but even in silicates containing little soda, the alteration of the outer flame is generally so slight that it cannot be perceived at all, or only indistinctly. Harkort's test also cannot be employed, because the amount of potassa is always comparatively small, and does not in the least alter the borax glass, coloured brown by the oxide of nickel.

On the other hand, according to Bunsen, the cobalt glass and indigo solution may be advantageously used to detect potassa in silicates also, by heating such compounds with gypsum, free from potassa and soda, in the flame; thus forming silicates of lime and sulphate of the alkali, which is volatile and colours the flame. Here, too, regard must be had to the remarks before made, if lithia is present (*vide* also remarks under lithia).

When not too trifling in its amount, potassa may also be with certainty detected in silicates by the wet way. About one hundred milligr. of the very fine powder are fused with one hundred milligr. each of borax and soda (previously ascertained to be perfectly free from potassa) to a transparent globule, free from bubbles. Should the assay contain considerable lime or magnesia and appear very infusible, somewhat more borax is added.* The fused globule is dissolved with dilute hydrochloric acid in a porcelain dish, evaporated to dryness, dissolved in a very little water, diluted with alcohol (not enough, however, to precipitate any salts), and filtered, or when clear, decanted from the residue of silica into a small test-tube, and the residue then washed with alcohol of 80°. If to the clear solution a few drops of a rather concentrated solution of bichloride of platinum are added, it will be at once seen whether potassa was present in the substance, and if so, whether in trifling or considerable quantity. When, for example, the volume of the double chloride is known, which separates when one hundred millgr. of a feldspar, yielding by chemical analysis, say fourteen per cent. of potassa are decomposed as above with soda and borax, an approximate estimate may be made of the amount of potassa in other silicates, from the quantity of the double chloride formed. This may be done with most certainty by employing a test-tube about ten millim. in diameter, drawn out below to a short tube two millim. in diameter, in which the crystalline chloride of potassium and platinum can settle, as shown in the cut.

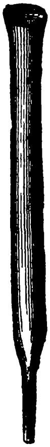
Fig. 64.

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* The amount of potassa derived from the burnt portion of an ordinary piece of charcoal is so exceedingly trifling that it could not be detected if the substance was before free from potassa, and therefore exerts no injurious influence upon the assay.

2. SODA, Na.

Its occurrence in the mineral kingdom.

Soda occurs quite frequently, but always is combination with other elements, being never found free.

a. It occurs as *chloride* ;
b. As *fluoride* ;
c. With *sulphuric acid* ;
d. With *nitric acid* ;
e. With *carbonic acid* and *water* ;
f. With *boracic acid* ;
g. With *silicic acid* in various silicates.

Examination for Soda.

Soda may be very readily detected in the natural salts above mentioned, as well as in any salt of which it forms a constituent, by heating quite a small portion of the salt in the loop of a platinum wire, with the tip of the blue flame. The outer flame immediately becomes enlarged and assumes a reddish-yellow tinge, even when a large amount of potassa or lithia is likewise present. If the salt is free from phosphoric or boracic acid and contains a very large amount of potassa, the flame is not pure reddish-yellow close to the assay, but rather violet, while at a greater distance the reddish-yellow colour alone prevails, and thus it may be quite well determined whether there is much less soda than potassa present. In presence of lithia the yellow flame is more strongly mixed with red, in proportion as the salt is richer in lithia and poorer in soda, so that with considerable lithia and little soda the flame is not reddish-yellow, but yellowish-red.

When a small quantity of any volatile salt of soda on platinum wire is brought within the zone of fusion of the gas-burner, the light proceeding from it renders a crystal of bichromate of potassa colourless when held near the flame. A better reaction is obtained when this deep red salt is replaced by a slip of paper, about one centim. square, coated with iodide of mercury, which assumes a white colour, with a shade of pale yellow. Potassa, lithia, and lime do not prevent this reaction.

According to Merz the soda flame appears orange-yellow through green glass, while the potassa flame is bluish-green and the lithia flame invisible.

In silicates, as well in the natural silicates as in other more or less fusible compound substances, the presence of soda can also be detected by the reddish-yellow flame, when small splinters are heated in the platinum forceps.

Bunsen's test, given above, is of especial interest in the examination of silicates, but can only be performed with the aid of the gas-burner. It is necessary to have a number of feldspars, accurately analyzed and arranged according to their increasing proportions of soda: these are ignited and kept in a pulverized state. If one of these and the specimen to be examined, with or without the addition of gypsum, are held in the zone of fusion at the same time, taking care that equal lengths of wire shall be heated, a strip of iodide of mercury-paper placed before the flame will be more or less blanched. If now the specimen to be determined is withdrawn from the flame and the paper assumes a perceptibly redder shade, the specimen contains more soda than the silicate used for comparison, while if the paper becomes whiter, the contrary is the case. By determining in this way between what numbers the reaction occurs, the amount of soda in the mineral under examination can be approximately ascertained. Various precautions must be observed in this test, but for these the reader is referred to the work cited above.

3. LITHIA, Li.

Its occurrence in the mineral kingdom.

Lithia is never met with in the free state, but always in combination with other bodies:

a. In combination with *phosphoric acid*, partly with and partly without fluorine;

b. In *silicates.*

Examination for Lithia.

Lithia is very readily detected in its salts by heating them, either on platinum wire or in the forceps, according to their fusibility, with the tip of the blue flame; a red tinge is always imparted to the outer flame, owing to the carmine-red colour which lithia communicates. Many strontia and lime salts, however, likewise give a red flame, which fact must be borne in mind, and the salt in question further tested, as will be directed under strontia and lime. The carmine-red produced by a pure lithia salt may appear quite different when other substances are present, which also colour the flame, *e.g.*, soda. Should the salt be free from soda, but contain phosphoric acid, which colours the flame bluish-green, the red and green do not unite, but both colours are separately visible. Triphylite shows this phenomenon.

A mixture of lithia and potassa salts upon a loop of platinum wire imparts to the flame a red tinge, which is less intense and more inclined to reddish-violet as the potassa increases in comparison with the lithia. A mixture of lithia, potassa, and soda salts in which lithia predominates, causes a yellowish-red flame, while if potassa predominates the flame is reddish-violet quite close to the assay, but reddish-yellow beyond. If the soda prevails and the assay is fused with the tip of the blue flame, the reaction of the potassa and lithia is concealed and only the reddish-yellow colour is perceptible, but by touching the salt with the outer flame only, and employing a very gentle blast, a distinct red flame may be frequently produced for a short time.

According to Stein, the lithia reaction is rendered indistinct and sometimes quite concealed by the soda because the temperature is too high. He has found that by fusing the assay on platinum wire, just so that it remains porous, and then soaking it in tallow and heating it in a candle flame, the red lithia flame is still distinctly visible, even when the amount of lithia is less than $\frac{1}{2000}$ that of the soda.

The effect of the lithia flame when viewed through cobalt glass or indigo solution has already been stated, and this affords a certain means of detecting lithia when mixed with soda and potassa. Cartmell recommends the observation through indigo solution, of the flame produced in the Bunsen burner by all these bases together by the side of a pure potassa flame. According to Bunsen, the distinction succeeds still better by observing *the successive alterations of the colour* which occur when each of the flames, produced side by side, is viewed through a stratum of indigo solution constantly increasing in thickness. For this purpose a prism of plate glass containing the solution is held before the eye. Pure carbonate of lithia, or chloride of lithium, shows a carmine-red flame through the thinnest stratum, while potassa still appears sky-blue to violet. The lithia flame grows feebler as the stratum increases in thickness, and disappears long before the thickest stratum comes before the eye. Potassa and soda have no influence in this case. Since the two salts just named give a more intense colour than any other lithia compounds, it is only necessary to mark that point of the prism where the colour imparted to the flame by these bodies becomes invisible, and then through the strata above this mark only potassa can be perceived, but lithia never. This portion of the prism then perfectly replaces a thick cobalt glass.

If now a portion of a potassa salt containing lithia is fused in the flame (soda alters the event but little, unless present in quite a large proportion), and the flame compared with a pure potassa flame produced opposite to it, the flame containing lithia appears redder than the pure potassa flame, through thin strata; through somewhat thicker strata the flames are equally red, if the amount of lithia is very trifling in comparison with the potassa; if the lithia predominates, the intensity of the now red lithia flame decreases perceptibly as the thickness of the stratum increases, while the pure potassa flame is scarcely weakened at all.

In silicates containing lithia this alkali may likewise be detected by the blowpipe flame. Unless there is too little lithia present it is at once recognized by the red flame produced by a small splinter heated in the forceps. In the absence of soda an intense purplish-red flame is produced while the assay is fusing; this is the case with lepidolite and castorite. Less intense and pure, but still distinctly perceptible colorations are produced by petalite and spodumene, when not too strongly heated.

Silicates containing only a little lithia, *e.g.*, lithia, tourmaline, and certain scapolites, colour the flame only very indistinctly, or not at all red. In this case Turner's method may be employed, which consists in making a paste of the finely-powdered mineral with a mixture of one part fluor spar, one and a half bisulphate of potassa, and a little water, and fusing it on a loop of platinum wire within the blue flame, at the same time carefully observing the colour of the outer flame. According to Merlet, two parts of the mixture must be taken for one of the silicate to make the reaction for lithia perfectly sure. If the silicate contains a little lithia, this colours the flame red, but not very intensely, the red inclining strongly to the violet of the potassa. If the silicate is free from lithia, only the violet potassa flame ensues; soda renders the reaction indistinct. If the silicate contains boracic acid, *e.g.*, tourmaline, a green flame is at first produced, showing the boracic acid, but afterward a more or less intense red flame is caused by the lithia.

The mixture proposed by Poole serves still better than Turner's for detecting lithia. It consists of two parts ignited gypsum and one part fluor spar.

Lithia may also be detected with certainty in silicates by using a Bunsen burner and the indigo prism. The assay powder is heated with gypsum in the zone of fusion, and opposite to it some carbonate of potassa, while both flames are observed through the prism, which is passed before the eye. If the assay contains lithia, its flame, at that point of the prism where the soda flame disappears, seems red in comparison with the cornflower-blue potassa flame. As the stratum increases in thickness the red lithia flame gradually loses its intensity, while the blue of the potassa flame passes through violet to red, which at a certain thickness of the stratum is quite similar to the colour of the lithia flame.

4. AMMONIA, NH^3.

Its occurrence in the mineral kingdom.

Ammonia is always found combined with other bodies.

a. As *ammonium* with *chlorine ;*
b. With *sulphuric acid ;*
c. With *phosphoric acid ;*
d. With *boracic acid ;*
e. With *carbonic acid.*

It is also found in trifling quantity as an unessential constituent of several other mineral substances, also in different kinds of clay, in certain native oxides of iron, and other mineral bodies, when they contain inclosed organic matter.

Examination for Ammonia.

In the compounds of ammonia, many of which can be recognized at once by their volatility in the matrass, the ammonia may be very easily detected by mixing a little of the substance with soda and gradually heating them in a matrass or closed tube over the spirit-lamp. An ammoniacal odour is evolved, and a bit of moistened red litmus-paper inserted in the tube is coloured blue. White clouds also form if a glass rod moistened with hydrochloric acid is held above the end of the open tube. As regards the other mineral substances in which the ammonia forms only an unessential and, frequently, only accidental ingredient, the ammonia may generally be recognized by testing them alone in the matrass, either at once by the odour, or by means of moistened red litmus-paper, since the ammonia escapes, whether it was present already as such, or was formed by heating a substance containing organic nitrogenous matters.

5. BARYTA, Ba.

Its occurrence in the mineral kingdom.

Baryta always occurs in combination with other bodies.

a. With *sulphuric acid ;*
b. With *carbonic acid ;*
c. With *silicic acid.*

Since barite sometimes forms an ingredient in ores dressed on a large scale, and is also added in many smelting processes, baryta frequently forms a constituent of slags, which also occasionally contain sulphide of barium.

Examination for Baryta,

Including the blowpipe characteristics of minerals containing baryta, the constituents of which can be for the most part detected at the same time.

SULPHATES.

a. Barite fuses only on the edge; colours the flame yellowish-green; on coal in the R. F. is reduced to sulphide. With soda on platinum foil it fuses to a clear mass; on charcoal it at first gives a clear bead with soda, which spreads out on continuing the blast, and sinks with ebullition into the coal as a strongly hepatic mass. This cut out, laid on silver foil, and moistened thoroughly, gives a black spot of sulphide of silver.

When the barite contains $Ca\ S$ and a little is treated with soda in powder, the sulphate of baryta and the soda with the sulphuric acid from the lime sink into the coal, leaving the lime, which generally adheres to the edges of the annual rings, and can be recognized by becoming quite luminous when treated for a while with the O. F.

b. Barytocelestite fuses with great difficulty, but more easily than barite, and colours the flame yellowish-green. The difference in the fusibility of the two can best be tested by pulverizing each with a little water, forming two thin crusts from the paste, and then testing one of these after the other at the same temperature. With soda it reacts like barite, as strontia also goes into the coal. The presence of strontia is detected by a special test, which will be given here for the sake of connection.

A little of the mineral is pulverized with some purified graphite and water in the mortar, dropped upon coal, and treated after careful drying for some time with the R. F. on both sides, the resulting compound of sulphides of barium and strontium is decomposed in a porcelain vessel with hydrochloric acid, the solution evaporated immediately to dryness, the salt dissolved in a few drops of distilled water, and then alcohol added until a s
setting fire to this and

the flame of the alcohol is coloured red with chloride of strontium. Even when the amount of strontia is trifling the colour may be distinctly seen if the solution of the salts in alcohol is absorbed by a ball of cotton wrapped about a loop of platinum wire and then set on fire. Here, as in the case of the blowpipe flame, care must be taken to avoid any impurities arising from soda salts, which would colour the flame intense reddish-yellow, and more or less conceal the red flame resulting from any trifling amount of strontia.

c. Dreelite fuses, according to Dufrenoy, to a white, blebby glass, and probably behaves with soda quite like barite containing lime.

CARBONATES.

a. Witherite fuses easily to a bead, colouring the flame distinctly yellowish-green and acquiring an alkaline reaction. With soda fuses very easily and goes into the coal. Dissolves in dilute hydrochloric acid with effervescence.

b. Barytocalcite is almost infusible, but produces an intensely yellowish-green flame. Strongly heated it *frits* on the surface, becomes bluish-green, and acquires an alkaline reaction. With soda the lime separates, while the rest sinks into the coal. With borax and S Ph (with addition of nitre), a feeble but distinct manganese reaction is obtained.

Bromlite behaves very similarly, with the exception of the manganese reaction.

The presence of lime in barytocalcite and bromlite can be detected by cautiously bringing the powdered minerals, moistened with hydrochloric acid and spread on a loop of platinum wire, near the tip of the blue flame. During the first action of the flame, distinct streaks of the yellowish-red lime flame are seen, which are, however, very soon concealed by the baryta.

SILICATES.

In silicates baryta cannot be detected, either by the flame, or by its behaviour with soda; nor is there any other means of determining it with certainty in the dry way. The wet way must therefore be used in connection with the dry method.

and *brewsterite* can be decomposed

by hydrochloric acid, so that, after filtering out the silicic acid all the bases may be detected. Upon adding bisulphate of potassa, or a few drops of sulphuric acid, to the solution, the baryta and strontia are precipitated as sulphates, and the only question is whether we have both of these salts, or which alone. To determine this the precipitate is thoroughly washed on a filter with hot water, spread on coal, dried with the blowpipe and then heated strongly until it adheres. The crust thus formed is tested in the forceps as to fusibility and the colour it imparts to the flame. If it fuses to a bead, and gives a red flame, it is strontia; if it fuses only with difficulty on the edges, and colours the flame yellowish-green, it is baryta; while if it is less fusible than in the former case and more fusible than in the latter, and at the same time produces rather a yellowish-green than a red flame, it is quite certain to be a mixture of both. In order to remove all doubtfulness from the reaction, it is, however, necessary to wash the precipitate so thoroughly that no soda can be present, as this would alter both the fusibility and the flame. If the yellowish-green flame has plainly shown baryta, but there is still a doubt whether strontia may not be present, the already tested assay, or a fresh portion of the precipitate, is to be treated with graphite on coal, exactly as directed for barytocelestite.

The manner in which the other bases are detected in harmotome and brewsterite, and the method of examining dressed ores and slags with the help of the wet way, is described in the examination of silicates containing lime, under lime.

According to Chapman, mixtures of $\dot{\mathrm{Ba}}\,\dot{\mathrm{S}}$ and $\dot{\mathrm{Sr}}\,\dot{\mathrm{S}}$ are fused in a platinum spoon with three to four parts of chloride of calcium, and the fused mass boiled with water. To the clear solution, diluted with water, a few drops of chromate of potassa in solution are added, and if a cloudiness is produced, this indicates the presence of baryta, since strontia is only precipitated from a concentrated solution.

MANGANESE ORES CONTAINING BARYTA.

In the three manganese ores, *braunite*, *hausmannite*, and *psilomelane*, the trifling amount of baryta can sometimes be detected simply by the feeble, yet distinct, baryta flame produced by a small splinter, either alone or moistened with hydrochloric acid. If this gives no satisfactory result, a not too small amount of the mineral is dissolved in hydrochloric acid, diluted with water, filtered if necessary, and tested with bisulphate of potassa or sulphuric acid. If a precipitate results, it can be collected on a filter and tested, as before described.

6. STRONTIA, Sr.

Its occurrence in the mineral kingdom.

Strontia occurs only :

a. With *sulphuric acid ;*

Also occasionally in trifling quantity in barite ;

b. With *carbonic acid ;*

c. With *silicic acid.*

Examination for Strontia,

Including the blowpipe characteristics of minerals containing strontia, the constituents of which can be for the most part detected at the same time.

SULPHATES.

Celestite (which generally decrepitates when crystallized) fuses to a milk-white bead, colouring the flame red. On coal in R. F. it spreads out and is changed to a difficultly fusible hepatic mass, consisting chiefly of sulphide. This treated with hydrochloric acid and alcohol, as directed under baryta, for minerals containing strontia, gives an intense red flame.

With soda fuses to a clear mass, which sinks into the coal with effervescence; any trifling admixtures of Ca or Fe are separated and behave as stated under baryta. The mass cut out from the coal gives a sulphur reaction on silver.

The method of detecting strontia in barytocelestite and certain barites has been given under baryta.

CARBONATES.

a. Strontianite swells in the blowpipe flame, putting forth ramifications which glow with a bright white light and fuse only on the thinnest edges; the flame is coloured red, and most strongly where the assay is most luminous. After ignition the assay reacts alkaline on red litmus-paper.

Strontianite dissolves with effervescence in dilute hydrochloric acid, and if the solution is evaporated to dryness and the resulting chloride of strontium treated with alcohol, or held in the blowpipe flame on platinum wire, it produces a red flame. According to Von Kobell, a bit of strontianite moistened with the acid and simply held in the candle flame will produce a red colour.

The purity of strontianite can readily be tested by fusion with soda. The pure mineral fuses to a clean mass and goes into the coal, but if any Ca, Fe, etc., are present, they are separated and left on the surface, as with barite.

b. Aragonite sometimes contains a little strontia, which can be detected by heating in the forceps a fragment of the assay, previously decrepitated in a matrass; it is infusible, but colours the flame more intensely red than an equally large piece of calcite. To detect it more certainly, a sufficient amount is dissolved in dilute hydrochloric acid and the strontia precipitated with a few drops of sulphuric acid. The precipitate is collected and treated as directed on p. 135. It fuses to a bead and colours the flame red like sulphate of strontia.

Strontia is detected in *brewsterite* as already directed, p. 135. Although strontianite sometimes occurs in ore-bearing veins, and it is therefore presumable that strontia also frequently forms a trifling constituent in dressed ores and the slags produced from them, it is quite certain that the very trifling amount could seldom be detected with certainty. When it is desired, however, to have regard to any strontia present in a slag or ore which is being examined for baryta, attention must be paid to the remarks made under baryta, p. 135.

7. LIME, Ca.

Its occurrence in the mineral kingdom.

Lime is of frequent occurrence and is found:

a. As *chloride ;*
b. As *fluoride ;*
c. With *sulphuric acid ;*
d. With *nitric acid ;*
e. With *phosphoric acid,* and at the same time as *calcium* with *fluorine* and *chlorine ;*
f. With *carbonic acid ;*
g. With *oxalic acid ;*
h. With *boracic acid ;*
i. With *arsenic acid ;*
k. With *tungstic acid ;*
l. With *antimonic acid ;*
m. With *hyponiobic acid ;*
n. With *titanic acid ;*
o. With *silicic acid* in a large number of silicates.

Calcite, dolomite, and fluorite being of very frequent occurrence in ore-bearing veins, lime often forms a considerable ingredient in dressed ores, especially when stamped dry, and consequently also in the slags resulting from smelting these ores; the amount of lime in the slags is also frequently increased by the addition of calciferous fluxes, necessary for smelting many ores. These slags occasionally contain Ca S and Ca F.

Examination for Lime,

Including the blowpipe characteristics of calciferous minerals, the constitutents of which can for the most part be discovered at the same time.

FLUORIDE OF CALCIUM AND ITS COMPOUNDS.

a. Fluorite frequently phosphoresces with a violet or greenish light, and generally decrepitates in the matrass. In the forceps it fuses to a bead, and after long heating colours the flame intense yellowish-red. On platinum foil and charcoal fuses with soda to a clear mass, which is opaque on cooling. With gypsum, barite, or celestite, it fuses easily on coal to a clear bead, opaque on cooling.

Dissolves very easily in considerable quantity in borax and S. Ph., but the glass becomes opaque when supersaturated.

Treated in the open tube with fused S. Ph., it evolves hydrofluoric acid (*vide* fluorine).

b. Yttrocerite (*vide* yttria).

SULPHATES.

a. Anhydrite yields no water, or only traces, in the matrass, while *gypsum* yields water and becomes milk-white.

B. B. both behave as follows: fuse with difficulty to an enamel-white bead and colour the flame red, but feebler than celestite. On coal in R. F. yield sulphide of calcium, which reacts alkaline on red litmus-paper and evolves a hepatic odour.

In borax on platinum wire dissolve to a clear glass, colourless unless oxide of iron is present, when the hot glass appears yellow. A supersaturated bead is opaque on cooling. If tested with borax on coal the saturated bead becomes yellow, owing less to a little iron than to the formation of sulphide of sodium. With soda on coal cannot be fused to a clear mass; distinction from barite and celestite. They are indeed decomposed, but the lime remains as an infusible mass, while the sulphate of soda and the excess of soda sink into the coal.

With fluorite they fuse to a clear bead, which is enamel-white on cooling and swells up and becomes infusible on continuing the blast.

b. Polyhalite, containing Ca S, Mg S, and K S, yields water and fuses on coal to an opaque red bead, which in the R. F. becomes white and forms a hollow crust, with a salt and somewhat hepatic taste. Fused on platinum wire a trifling amount of Na Cl causes a soda flame, so that the potassa can only be seen by using cobalt glass or indigo solution (*vide* potassa).

In borax dissolves easily with effervescence to a clear bead, somewhat coloured with iron, which is opaque when very much is added. In S. Ph. dissolves easily to a clear, colourless bead, opaque on cooling, which shows no iron colour unless a great deal is added. With soda it is decomposed, yielding an earthy mass, which in the R. F. is yellowish from Na S. With fluorite fuses to an opaque bead.

Lime and magnesia can only be separated by the wet way, and each tested by itself with the blowpipe. The mineral is dissolved in dilute hydrochloric acid, a trifling amount of sesquioxide of iron precipitated by ammonia, and then the lime precipitated with oxalic acid and the magnesia with salt of phosphorus.

c. Glauberite, containing besides Ca S, some Na S, which is recognized by the soda flame (*vide* soda), decrepitates in the matrass with some violence and yields very little water; at incipient redness it fuses to a clear mass, yielding nothing volatile. On coal becomes at first white, then fuses to a clear bead, opaque on cooling. In the R. F. the bead becomes infusible and hepatic, and after blowing longer the Na S goes into the coal and leaves the lime behind. In borax and S. Ph. dissolves in large quantity with effervescence to a glass which is opaque on cooling. It is decomposed with soda on coal to a hepatic mass, which sinks in and leaves the lime. With fluorite fuses like gypsum.

d. Dreelite probably reacts like a calciferous barite with soda. The lime can be detected with certainty by digesting a little of the powdered mineral with dilute hydrochloric acid, adding water, filtering out the residue of sulphate of baryta, and precipitating the lime with oxalic acid after making the filtrate ammoniacal.

NITRATE OF LIME.

In the matrass yields water, and when strongly heated nitrous acid. On platinum wire gives a strongly luminous mass, which colours the flame yellowish-red. Deflagrates slightly on coal, leaving a white, earthy, alkaline mass, which does not sink into the coal with soda.

PHOSPHATE OF LIME WITH CHLORIDE AND FLUORIDE OF CALCIUM.

Apatite sometimes phosphoresces in the matrass. In the forceps fuses with difficulty on the edge to a translucent glass, not colouring the flame distinctly. The fine powder moistened with sulphuric acid produces a transient bluish-green flame. The phosphoric acid can also be otherwise detected (*vide* phosphoric acid). In borax dissolves slowly to a clear glass, frequently yellow from iron while hot, and which can be made opaque by flaming with a certain degree of saturation; when more is added it becomes opaque of itself on cooling. In S. Ph. dissolves largely to a clear glass, which when nearly saturated becomes opaque and shows crystalline facets on cooling; these are less distinct than those produced by phosphate of lead in this salt. A fully saturated glass becomes milk-white without showing facets. With equal parts of soda swells with effervescence to an infusible mass, more soda goes into the coal. Any manganese present can be detected with soda and nitre on platinum foil (*vide* manganese). Chlorine and fluorine, if not in too small quantities, are found by the tests given elsewhere.

To ascertain further the presence of lime, the powdered mineral is dissolved in hydrochloric acid, a few drops of sulphuric acid are added, the acid solution diluted with three volumes of strong alcohol, and shaken. Sulphate of lime separates and can soon be filtered off: after being washed with alcohol it must react B. B. like gypsum. After removing the alcohol by evaporation, other ingredients like alumina and oxide of iron can be tested for. (The above solution cannot first be treated with ammonia, for the lime would go down as phosphate again.) To test the apatite for magnesia also, it must be fused in powder with soda and silicic acid, as will be given under the phosphoric acid, the mass treated with water, the carbonates of the earths and any residual silica dissolved in hydrochloric acid, the solution diluted with water, and the silicic acid, with any traces of alumina and sesquioxide of iron, precipitated with ammonia; after which the lime is thrown down with oxalic acid and the magnesia with salt of phosphorus.

Among the remaining phosphates of lime, *hydroapatite, pyroclasite*, and *glaubapatite* yield water; fluorine and chlorine can only be detected in hydroapatite. The remaining reactions are like apatite.

CARBONATES.

a. Calcite. Decrepitates sometimes in the matrass, and when containing metallic oxides changes in colour. In the forceps is infusible, becomes caustic, feebly luminous, and colours the flame red, but far more feebly than strontianite. When afterward moistened with hydrochloric acid it gives a distinct characteristic lime flame. After thorough ignition it has an alkaline reaction. Dissolves with effervescence in the glass fluxes and reacts like lime; if it contains metallic oxides they can be at the same time recognized, and then traces of manganese must be specially tested for (*vide* manganese). With soda on platinum foil fuses to a clear mass, while the metallic oxides are separated; on coal fuses at first, but afterward most of the soda sinks in, leaving an infusible residue, which is luminous under a strong blast. Sometimes a sulphur reaction is obtained on silver foil.

b. Aragonite crumbles to pieces B. B.; otherwise reacts like calcite, but when containing strontia gives a more intense red flame, and when containing lead does not give a pure red but a bluish flame, while with soda on coal in the R. F. it deposits a slight lead coat. This is the case with *tarnovicite.*

c. Gay-Lussite yields water in the matrass, and then has an alkaline reaction. In the forceps fuses to an opaque bead, and gives a strong soda flame. With the fluxes and soda reacts like carbonate of lime.

d. Plumbocalcite reacts like plumbiferous aragonite, but gives a stronger lead coat.

e. Predazzite, pencatite, hydrodolomite, dolomite, and all compounds of carbonates of lime and magnesia, react like carbonate of lime without magnesia, except that with soda on platinum foil they do not melt to a clear mass, because the magnesia is separated and can thus be detected. If the reaction is not distinct enough, the wet way must be employed (*vide* magnesia). According to Von Zehmen lime may be distinguished from dolomite thus: the doubtful mineral is powdered as finely as possible, placed in a slight hollow on platinum foil, and heated over the spirit-lamp several minutes, until it glows through and through. Lime forms after ignition a slightly coherent mass, which is not entirely broken up when carefully thrown from the foil; it also shows a tendency to adhere to the foil, and can only be separated by gentle concussion. Dolomite powder when ignited is not coherent, but falls as a loose powder from the platinum; with many dolomites there is a lively motion during the ignition, caused by the rapid evolution of carbonic acid.

Oxalate of lime, whewellite, is converted into carbonate by feeble ignition, and its blowpipe reactions then resemble those of calcite.

BORATES.

Borocalcite, hydroborocalcite, and *hydroboracite* yield much water, and fuse in the forceps with slight intumescence to a clear glass; the first and third colour the flame pale greenish, the second reddish-yellow; moistened with sulphuric acid, all three produce the yellowish-green boracic acid flame. Dissolve largely in soda and S. Ph. to a clear glass; also on coal with a little soda.

Lime and magnesia can only be separated in the wet way, by dissolving the powdered mineral in hydrochloric acid, diluting the acid solution with water, and adding soda enough to combine all the boracic acid with itself, so that no borate of magnesia may be thrown down on adding ammonia. The solution, with free hydrochloric acid, is then treated with ammonia in excess, and the lime and magnesia successively precipitated with oxalic acid and salt of phosphorus.

Rhodizite yields no water; fuses only on the edges, colouring the flame green. The lime is detected as above.

ARSENATES.

a. Haidingerite, pharmacolite, and *picropharmacolite* behave similarly. In the matrass yield much water, especially the latter. They also become opaque, and if a fragment is afterward treated in the forceps it fuses with intumescence to a white enamel, giving a light blue arsenic flame. On coal in the R. F. fuse to a semi-transparent bead, sometimes bluish from cobalt, and evolve an arsenical odour. In borax and S. Ph. like lime, but on coal the arsenic acid reduces and evolves arsenical fumes.

Decomposed with soda on coal, evolving arsenical fumes, while the soda sinks in and leaves the lime. If the residue from picropharmacolite is dissolved in hydrochloric acid, the solution supersaturated with ammonia, and the lime precipitated with oxalic acid, the magnesia can be detected with salt of phosphorus.

b. Berzeliite is infusible and becomes grey (giving probably a light-blue flame). With the fluxes like the above minerals, but colours the borax bead distinctly with manganese. To detect the magnesia the crust left by soda on coal should be dissolved in hydrochloric acid, diluted with water, and the lime, magnesia, and manganese precipitated as described under the silicates.

TUNGSTATE OF LIME.

Scheelite fuses on the edge to a semi-transparent glass, not colouring the flame. In borax in O. F. dissolves easily to a clear glass, which soon becomes milk-white and crystalline, and cannot be coloured in the R. F., even on coal with tin. In S. Ph. dissolves easily in the O. F. to a clear bead, colourless only when quite free from iron, which treated in the R. F. becomes blue from the formation of binoxide on cooling. Varieties containing iron give a brownish glass in the R. F., which only becomes blue when treated with tin on charcoal. Tested with fused S. Ph. in the open tube gives a little hydrofluoric acid. When a little of the powdered mineral is fused with four to five parts of soda in the platinum spoon and then dissolved in hot water, tungstate of soda and soda dissolve, leaving lime and a little sesquioxide of iron and manganese, which may be tested B. B. The manner of separating the tungstic acid will be given under tungsten; as well as another simple method of detecting tungstic acid in combination.

Antimonate of lime, romeite.—With soda on coal the antimonic acid is reduced and volatilizes, forming a white coat, while the lime remains and the excess of soda sinks into the coal.

HYPONIBOATES.

a. Pyrochlore from Miask yields only traces of water. In the forceps infusible, but becomes yellow, and gives a yellow flame mixed with much red (soda and lithia). With borax in the O. F. a clear glass, reddish-yellow when hot, colourless when cold; saturated to a certain degree, it becomes opaque and reddish-gray by flaming; with more it becomes opaque of itself on cooling, and is yellowish to reddish-gray. Dissolves easily in S. Ph. in the O. F. to a clear yellow glass, which in the R. F. becomes dark brownish-red, as if from ferriferous titanic acid; with tin the glass becomes violet. It is therefore free from uranium. No manganese reaction can be obtained.

To detect lime in pyrochlore a little of the powder is fused with six to eight parts of bisulphate of potassa in the platinum spoon, the fused mass poured out, pulverized, and treated with water, when the basic constituents are mostly dissolved and the hyponiobic acid remains, mixed with part of the titanic acid and some gypsum. After filtering and washing for a long time, so as to separate the gypsum from the acids named, a few drops of nitric acid are added to the filtrate, which is heated to boiling, and then the dissolved titanic acid separates as a white powder. After filtering this off, adding some hydrochloric acid and then ammonia, and again filtering out the precipitated earths and metallic oxides, the lime can be thrown down with oxalic acid and tested B. B.

In determining the other constituents the directions must be followed which are given under yttria, in the case of tantalates, etc., that must first be fused with bisulphate of potassa.

TITANAIE OF LIME.

Perofskite. According to G. Rose it behaves as follows:

In the forceps and on coal infusible. Dissolves largely in borax to a clear greenish glass, colourless when cold. In the R. F., when slightly saturated and warm, light yellowish-green, on cooling clear as water; more strongly saturated it is brown on cooling.

With S. Ph. in O. F. same as with borax; in R. F. grayish-green, but becoming more or less violet-blue, according to the amount dissolved.

To detect the lime the mineral is treated with bisulphate of potassa, as described for pyrochlore.

SILICATES.

The silicates as well as the calciferous silicates when varying in their composition, can be distinguished from one another by the blowpipe and with reagents; the presence of lime can also frequently be ascertained from the blowpipe characteristics. The following indications are to be observed: 1. Tumefaction and bubbling during the test for fusibility, often seen in calciferous silicates. 2. Their behaviour with borax and S. Ph.; since most of these dissolve easily in borax, while with S. Ph. not only is the silicic acid separated, but the glass also generally becomes opalescent on cooling. 3. Their behaviour with soda; they fuse to a bead with not too much soda; with more they sometimes give a slaggy mass.

To determine the lime with certainty in silicates not perfectly decomposed by hydrochloric acid, they must be fused with soda and borax, and if necessary, silver; or, in case of slags containing many metallic oxides, with gold, on coal to a bead, which is pulverized and treated with hydrochloric acid and water. The solution filtered off from the silica is tested, after converting any protoxide of iron into sesquioxide by a few drops of nitric acid, first with a drop of dilute sulphuric acid, or a little bisulphate of potassa, for baryta, and then ammonia is added in slight excess to precipitate any alumina and sesquioxide of iron and chromium. The method of separating alumina from the other two oxides will be given under silicates of alumina. When tungstic acid is present, as in certain tin slags, the silica must be tested for it, according to the directions given for silicates under tungsten. The lime is then found in the ammoniacal solution, which may also contain magnesia and protoxides of manganese and cobalt, in the following way:

When the tests with fluxes have shown no manganese or cobalt, or only a very little, the lime is at once thrown down with oxalic acid, and the oxalate after settling is filtered out, washed, and tested B. B. if desired. The filtrate, to which a little oxalic acid is added to make certain that all lime is separated, is treated with S. Ph., dissolved in a little water, which precipitates magnesia and protoxide of manganese as phosphates in combination with ammonia. This precipitate is washed on a filter with cold water containing a little carbonate of ammonia, and tested B. B. On coal it fuses to a bead, enamel-white unless cobalt is present, when it is blue or violet. By fusing this powdered bead with soda and nitre on platinum foil the presence of any manganese is speedily ascertained (*vide* manganese). The protoxide of manganese becomes more highly oxidized and combines with the alkalies to a fluid mass, which can be driven about with the flame, while the magnesia adheres firmly to the foil and can be distinctly seen. The cold mass is bluish-green if manganese is present, but otherwise white.

If, as is rarely the case, the tests with the glass fluxes have shown much manganese or cobalt, and it is desired to determine the presence of magnesia with certainty after separating the lime, this must be done as follows: To the ammoniacal solution sufficient sulphide of ammonium is added to precipitate the manganese and cobalt, which are filtered off and washed with water containing a little sulphide of ammonium. The precipitated sulphides may be roasted on coal and then tested with S. Ph. for cobalt, and with soda and nitre for manganese. The filtrate, not too much diluted with wash-water, is then acidified with hydrochloric acid and heated until it has no odour of sulphuretted hydrogen and appears clear, while the separated sulphur has settled. After then filtering out the sulphur and adding an excess of ammonia, the lime is precipitated with oxalic acid and the magnesia with S. Ph.

The method of decomposing silicates containing besides lime, yttria, zirconia, and oxide of cerium, will be given under yttria and zirconia.

Datolite and *botryolite*, containing boracic acid as a base, behave as follows:

In the matrass yield water. In the forceps swell up and fuse rather easily to a dense, clear, generally colourless glass;

colouring the flame distinctly green from boracic acid. The flame becomes more intensely green when the dehydrated assay is moistened with sulphuric acid. Dissolve easily in borax to a clear glass, sometimes showing a very little iron. Dissolve in S. Ph., leaving a silica skeleton; the strongly saturated glass is opaque to enamel-white on cooling.

To separate the lime from the boracic acid, the powdered silicates are decomposed by hydrochloric acid, and the gelatinous silica filtered out after dilution with water, when the solution is supersaturated with ammonia and the lime precipitated with oxalic acid and tested B. B. Magnesia, if present, would be thrown down as borate on adding ammonia, since there is neither soda nor potassa in the solution.

The silicates containing titanic acid, viz., *Titanite, guarinite, schorlomite,* and *keilhauite* behave as follows:

Titanite in the matrass sometimes yields traces of mechanically combined water. The yellow variety is unaltered, the brown becomes yellow, and according to Berzelius a variety from Frugard in Finland glows like gadolinite, but far more feebly. In the forceps it fuses with some intumescence on the edge to a blackish glass, without colouring the flame. Dissolves easily in borax to a clear yellow glass. Dissolves with difficulty in S. Ph., the undissolved portion becoming milk-white. On coal with tin the bead becomes pale reddish-violet, showing titanic acid, and the glass then generally opalesces on cooling.

Upon fusing a little of the powdered mineral with eight parts of bisulphate of potassa in the platinum spoon, and treating the fused mass at a temperature of 65° to 75° C. with a sufficient amount of water, to which a few drops of hydrochloric acid have been added to facilitate the solution of the sulphate of lime, this and the titanic acid dissolve, leaving the silica behind. After filtering the latter out and washing it, a few drops of nitric acid are added to the filtrate, which is heated to boiling, and then, if the solution is dilute enough, the titanic acid separates and can be collected on a filter and tested B. B. The filtrate from the titanic acid is saturated with ammonia to precipitate sesquioxide of iron, and then the lime can be thrown down by oxalic acid.

Schorlomite. In the forceps fuses with difficulty on the edge; the borax bead is yellow in the O. F., green in the R. F.; the S. Ph. bead, with tin in the R. F., becomes violet. Rammelsberg.

Keilhauite. In the forceps is infusible and changes little or not at all in colour; with the fluxes like titanite, but shows a considerable amount of iron.

The wet way must be employed to detect the constituents that cannot be recognized with the blowpipe. The mineral is fused as directed for titanite with bisulphate of potassa, etc., and after separating the titanic acid from the solution, Y, Al, and Fe are thrown down, leaving Ca, Mg, and Mn in solution. The latter are precipitated by oxalic acid and S. Ph.; the separation of Y from Fe and Al will be given under yttria.

8. MAGNESIA, Mg.

Its occurrence in the mineral kingdom.

Magnesia forms a more or less essential constituent in many mineral substances.

a. As *free magnesia;*
b. In combination with *water;*
c. In combination with *metallic oxides;*
d. As *magnesium* with *chlorine;*
e. With *sulphuric acid;*
f. With *phosphoric acid;*
g. With *carbonic acid;*
h. With *boracic acid;*
i. With *arsenic acid;*
k. With *silicic acid* in many *silicates.*

As minerals containing magnesia sometimes occur in the gangue of ores, more or less magnesia is also to be found in these ores, especially when stamped dry, and in the slags obtained from smelting them.

Examination for Magnesia.

Including the blowpipe characteristics of magnesia minerals, the constituents of which can, for the most part, be ascertained at the same time.

MAGNESIA AND ITS HYDRATE.

Periclasite and *brucite* (*nemalite*), the latter of which yields considerable water, react alkaline after strong ignition, and behave with fluxes and cobalt solution like magnesia, but sometimes colour the glass more or less with iron.

Magnesioferrite gives a strong iron reaction with fluxes, and its copper is detected by moistening it with hydrochloric acid and heating it in the O. F.; the magnesia can only be found in the wet way with S. Ph. after precipitating the iron with ammonia.

CHLORIDES AND SULPHATES.

a. Tachydrite yields much water and fuses easily. Magnesia can only be found in the wet way after separating the lime (see lime) and adding S. Ph. The chlorine can be detected by means of a bead of S. Ph. containing oxide of copper (*vide* chlorine).

b. Carnallite yields much water and gives a strong soda flame. The potassa can, however, be easily seen by using cobalt glass or indigo solution (*vide* potassa). On coal in the R. F. with soda a mass is obtained which reacts for sulphur on silver foil; with a bead of S. Ph. containing oxide of copper a chlorine reaction. The presence of the various earths can only be shown by the wet way.

c. Kieserite and *epsomite* yield in the matrass water, which, after long heating, has an acid reaction. On coal lose water and sulphuric acid, becoming luminous, infusible, and alkaline. The Na Cl in kieserite causes a reddish-yellow flame. They behave with soda and S. Ph. like magnesia, and with soda on coal swell, but do not fuse; the mass moistened with water evolves a hepatic odour. With cobalt solution they become rose-red.

d. Bloedite when heated loses water rapidly; at a red heat fuses quietly to a transparent globule, white on cooling. (Dana.)

The artificial salt behaves similarly, giving a soda flame, and when strongly heated on coal it boils, spreads out somewhat, and leaves an infusible mass, yellow from sulphide of sodium, while most of the sulphate of soda is absorbed. The mass gives a strong sulphur reaction on silver.

e. Pickeringite and *bosjemanite* fuse in the matrass with their water of crystallization, froth, and yield water. The dry mass strongly heated evolves sulphurous acid. Both dissolve perfectly in borax and S. Ph., and bosjemanite imparts a manganese colour, especially to borax. The magnesia can only be certainly detected by dissolving a little of the mineral in water, acidifying the solution with hydrochloric acid, precipitating alumina with ammonia, and then separating the manganese from the ammoniacal solution with sulphide of ammonia, after which the magnesia can be thrown down by S. Ph., as directed under lime.

PHOSPHATES.

Wagnerite fuses only in thin splinters with great difficulty, with formation of a few bubbles, to a dark greenish-gray glass. Moistened with sulphuric acid, it momentarily imparts a pale bluish-green tinge of phosphoric acid to the flame. Dissolves easily in soda and S. Ph. to a clear glass, slightly yellow from iron. Fuses together with soda with effervescence, but is not dissolved; on platinum foil a weak manganese reaction. Treated in an open glass tube with fused S. Ph. it yields hydrofluoric acid (*vide* fluorine).

To detect the magnesia with certainty a small portion of the very finely-powdered mineral is fused with about three parts of a mixture of soda and hydrate of potassa in equal quantities in the platinum spoon, and then treated with water in a porcelain vessel over the lamp. Phosphate of soda and potassa dissolve, with any silica present and the excess of carbonate of soda, while the magnesia and a little sesquioxide of iron remain behind. These are washed on a filter and dissolved upon it with a little hydrochloric acid and the solution collected in a test tube, in which the iron is precipitated with ammonia, and then any lime present is thrown down with oxalic acid, after which the magnesia can be separated from the ammoniacal solution by S. Ph. and tested B. B. The phosphoric acid can also be found in the first filtrate, as will be described under phosphoric acid.

Lazulite in the matrass yields a little water and loses its blue colour. B. B. swells up, cracks, crumbles to pieces, and becomes white, but does not fuse. It colours the flame pale bluish-green, from phosphoric acid, and more intensely when moistened with sulphuric acid. Dissolves in borax to a clear glass, coloured yellow by iron while hot. In S. Ph. becomes transparent on the edges, and gradually dissolves to a clear glass, showing iron while warm. With soda on coal swells, yielding an infusible mass, and with cobalt solution assumes a fine blue colour.

The magnesia can be found in these minerals, which also contain phosphate of alumina, by fusing the mineral on coal with soda and silicic acid in the O. F. and treating the fused bead as will be directed under phosphoric acid. On treating the assay with water there remains, besides the magnesia and sesquioxide of iron, silicate of alumina and soda; after filtering out and washing this residue it is dissolved on the filter in hydrochloric acid, and then the alumina, sesquioxide of iron, and silica can be precipitated with ammonia, any lime present with oxalic acid, and the magnesia with S. Ph. The precipitate formed by the ammonia is dissolved, if desirable in hydrochloric acid, evaporated to dryness, the mass dissolved again in water, and the silica filtered out, after which the alumina and iron are thrown down together by ammonia and separated with a solution of potassa, as will be directed for the examination of silicates under alumina.

As these minerals contain trifling amounts of silica which could not be found at the same time, the following method may be employed: the powdered mineral is first fused with soda and borax on coal, and the mass treated with hydrochloric acid, etc. The solution filtered off from the silica is treated with an excess of ammonia, and S. Ph. is added for the sake of certainty, so that all the bases may be precipitated in combination with phosphoric acid. The resulting precipitate is filtered off, washed, dried, fused with soda and silica on coal, treated with water, and the residue further examined as has already been directed.

CARBONATES, HYDROUS AND ANHYDROUS.

Magnesite yields very little or no water. B. B. infusible, but shrinks somewhat and acquires an alkaline reaction.

Hydromagnesite and *lancasterite* yield water in the matrass and then behave like magnesite. With the fluxes, soda, and cobalt solution, the three minerals behave like carbonate of magnesia. When some Mg is replaced by Fe or Mn as in *breunerite, mesitite, pistomesite, sideroplesite,* etc., the fluxes show a manganese or iron reaction, and the cobalt solution test must frequently be given up. When, moreover, a part of the magnesia is replaced by lime, as in *dolomite, ankerite, predazzite, pencatite, hydrodolomite,* etc., the blowpipe reaction for magnesia becomes quite uncertain, and the wet way must be brought to aid it. A little of the powder is dissolved in hydrochloric acid, which it does easily with effervescence when warmed, the iron converted into perchloride by boiling with a few drops of nitric acid and then precipitated with ammonia in excess, the lime separated with oxalic acid, and the magnesia, with the frequently small quantity of protoxide of manganese, thrown down by S. Ph., as a precipitate which can be tested with cobalt solution.

BORATE.

Boracite is unaltered in the matrass and yields no water, or only traces. B. B. fuses with intumescence to a nearly white, crystalline bead, and gives a green, boracic acid flame. Dissolves easily in borax to a clear glass, yellow with iron while hot. In S. Ph. also dissolves easily, but the somewhat saturated glass can be made opaque by flaming, and when supersaturated becomes opaque of itself on cooling. It combines very easily with soda, yielding with just the right amount a clear glass, which on cooling crystallizes with facets like phosphate of lead. With more soda a clear glass, not crystallizable, and which may be regarded as borax-glass containing magnesia. The powdered mineral heated on coal with oxide of copper gives a momentary flame of chloride of copper (*vide* chlorine). To detect the magnesia and establish the absence of other earthy bases, a little is dissolved in hydrochloric acid and further treated as for *borocalcite*.

Stassfurtite reacts like boracite, but yields a little water.

SILICATES.

The behaviour of the silicates and the silicates containing magnesia, after being tested as to fusibility and the presence or absence of water, is in general as follows: with borax they dissolve more or less easily to a clear glass which is more or less yellow if iron is present. With S. Ph. they dissolve, leaving a skeleton of the silica; those containing little or no alumina most easily; the glass generally opalesces on cooling.

They also behave dissimilarly with soda; most of them, however, fuse with a little soda to a bead, and give a slag-like mass with more.

Cobalt solution produces a magnesia reaction only with silicates containing few or no colouring metallic oxides, and also not too much alumina, since, *e. g.*, a notable amount of sesquioxide of iron entirely conceals the red colour assumed by magnesia with cobalt solution, while if the silicate contains much alumina without colouring oxides, the solution produces rather a blue than a red colour, although when the amount of alumina is not very great this appears nearly violet, from the blue of the alumina with the rose-red of the magnesia.

When the magnesia cannot be detected with the blowpipe, silicates, as well as dressed ores and slags, must be examined for it by the method specially described under lime.

SILICATES WITH PHOSPHATES.

Sordavalite yields water and fuses quietly to a black bead in the forceps or on coal (Berzelius).

Dissolves easily in borax to a glass coloured yellow with iron (green, Dana), with S. Ph. decomposed, leaving a silica skeleton.

The magnesia is found by fusing the mineral with some soda on coal in O. F. to a bead, which is pulverized and treated with water. Silicate and phosphate of soda dissolve and the bases remain with some silica. After dissolving this residue in hydrochloric acid, and separating iron and alumina with ammonia, the magnesia is precipitated with S. Ph.

SILICATE WITH FLUORIDE.

Chondrodite sometimes yields traces of water and turns black (C. from Vesuvius is unaltered in the matrass), but in the open flame again becomes white, does not fuse, and when free from iron assumes a milk-white appearance. In an open tube yields a sery distinct fluorine reaction, both alone and with fused S. Ph. (*vide* fluorine). Dissolves slowly in borax to a clear glass, sometimes coloured with iron, which can be made opaque by flaming when strongly saturated, and then appears more or less crystalline. Decomposed by S. Ph., leaving a silica skeleton; the glass opalesces on cooling. With a little soda forms a difficultly fusible gray slag; with more swells and becomes infusible.

When free from iron gives a pale rose-colour with cobalt solution; when containing iron a grayish-brown colour. In the latter case the magnesia can only be determined with certainty by the aid of the wet way.

ALUMINATES.

a. Spinel alone is unaltered. The red variety from Ceylon becomes black and opaque in the forceps, but in cooling becomes translucent and chrome-green, then nearly colourless, and finally red again. Dissolved slowly by borax, even in powder, to a clear, feebly yellowish-green glass. In powder is dissolved rather easily by S. Ph. to a clear glass, reddish while warm, but when cold feebly, though distinctly, chrome-green. With soda and nitre on platinum foil shows traces of manganese. The fine powder assumes a blue colour with cobalt solution.

The magnesia can only be detected with certainty by fusing the very fine powder with two volumes of soda and three of borax on coal to a bead, which is pulverized and treated with hydrochloric acid.

b. Hydrotalcite (*völknerite*) in the closed tube yields much water. B. B. infusible, but exfoliates somewhat, and gives out light. A weak rose-red with cobalt solution. With the fluxes intumesces and gives a clear, colourless glass. The Snarum mineral reacts for iron. (Dana.)

c. Ceylonite, hercynite. Change colour when heated, but are infusible. Dissolve in borax and S. Ph. to a clear glass, coloured by iron. The magnesia can only be found as with spinel.

d. Chlorospinel behaves like ceylonite, but a copper reaction is obtained by means of S. Ph. with tin on charcoal.

By fusing the powdered mineral on coal in the R. F. with soda and borax beside a silver button, until all the copper is reduced and united with the silver and the bead is quite transparent, the magnesia can be very easily found as directed for spinel.

9. ALUMINA, $\ddot{\mathrm{Al}}$.

Its occurrence in the mineral kingdom.

Alumina occurs very extensively:

a. As *aluminium* with *fluorine* and *fluoride of sodium;*
b. In a *free state;*
c. Combined with *water of hydration;*
d. With *sulphuric acid;*
e. With *phosphoric acid;*
f. With *mellitic acid;*
g. With *silicic acid* in many silicates;
h. As an acid.

Since most of the rocks in which ore-bearing veins occur contain more or less alumina, and since in extracting the ores it is seldom possible perfectly to separate the adherent rock and the alumina-bearing minerals occurring in the vein itself, it follows that alumina frequently forms a notable constituent of ores dressed in the dry way and also in the slags produced in smelting them.

Examination for Alumina.

Including the blowpipe characteristics of alumina-bearing minerals, the constituents of which can for the most part be ascertained at the same time.

FLUORIDE OF SODIUM WITH FLUORIDE OF ALUMINUM.

a. Cryolite sometimes decrepitates in the matrass.

In the open tube, heated so that the flame enters the tube, much hydrofluoric acid is evolved, which attacks the glass, while the condensed water in the tube reacts acid with Brazil-wood paper. B. B. fuses very easily, yielding part of its fluorine and giving a strong soda flame. On coal fuses very easily to a clear bead, opaque on cooling. After blowing longer the bead spreads, the fluoride of sodium goes into the coal, and a suffocating odour of hydrofluoric acid is perceptible, while a crust of alumina remains, which assumes a blue colour with cobalt solution.

Dissolves largely and easily in borax and S. Ph. to a clear glass, milk-white on cooling. Fuses with soda to a clear glass, which on cooling spreads out and becomes milk-white.

b. Chiolite yields no water, fuses very easily, and behaves otherwise like cryolite.

ALUMINA.

Corundum alone is quite unaltered. In borax dissolves with difficulty, but perfectly, to a clear glass, colourless if no iron is present. With S. Ph. dissolves only in powder, and slowly, to a clear glass, and is not attacked by soda. The very finely-powdered mineral assumes a fine blue colour with cobalt solution.

When it is desired to examine it for Si, $\rlap{-}\text{F}\text{e}$, $\dot{\text{C}}\text{a}$, etc., it should be finely pulverized in the steel mortar, to avoid any $\ddot{\text{S}}\text{i}$ from the agate mortar, fused on charcoal with soda and borax, and further treated with hydrochloric acid. According to H. Rose, on fusion with bisulphate of potassa it readily yields a mass soluble in water.

HYDRATE OF ALUMINA.

a. Diaspore yields a little water at first and more when heated to glowing. It decrepitates little, or not at all; Berzelius, however, examined a diaspore from an unknown locality, which decrepitated with violence, crumbling into lustrous, white scales, and only yielding water at a red heat.

B. B. it is infusible. With the fluxes like alumina, and if notably ferruginous the borax bead is yellow. Finely powdered it assumes a blue colour with cobalt solution.

b. Gibbsite behaves like diaspore; when containing phosphoric acid it colours the flame pale green.

SULPHATES.

a. Felsobanyite, aluminite, and *alumian.* The two former yield in the matrass much water, and at a high temperature sulphurous and sulphuric acids, recognizable by the odour and with litmus-paper. With borax and S. Ph. like alumina. With soda an infusible hepatic mass, and with cobalt solution a fine blue. *Alumian* behaves similarly, but yields no water.

b. Alunite yields water and sometimes crumbles to powder, especially when crystallized. More strongly heated, a little sulphate of ammonia is sometimes sublimed, while sulphurous and sulphuric acids are evolved and render the glass cloudy. In the forceps gives a violet flame, becoming reddish-yellow further from the assay. If the soda reaction is too strong, the potassa is easily found. (See Examination for Potassa.)

Dissolves easily in borax and S. Ph. to a clear, colourless glass, but sometimes leaves a silica skeleton in the latter. Is infusible with soda, but gives on coal a hepatic mass. With cobalt solution a fine blue.

c. Alunogen swells up in the matrass, yielding much water, and at a higher temperature sulphurous and sulphuric acids. The dehydrated salt is infusible B. B., but frequently gives a soda or potassa flame. With fluxes like aluminite, but the borax bead is frequently yellow from iron. Gives a fine blue with cobalt solution, unless containing too much iron. The salt is easily soluble in water, and therefore any other earths present can be readily found by proper reagents.

d. Kalinite fuses in its water of crystallization and froths, yielding much water; the residue heated to redness evolves sulphurous and sulphuric acids. The dehydrated alum is infusible B. B. and gives a potassa flame; if this is prevented by soda, the potassa is found. (See, as before, Potassa.) With the fluxes and cobalt solution like alunogen.

e. Mendozite behaves like kalinite, but only gives a soda flame, in which no potassa can be detected.

f. Tschermigite at first acts like the preceding two in the matrass, but with a stronger heat some sulphate of ammonia is sublimed. With the fluxes, etc., like kalinite. Mixed with soda and gently heated in the matrass, carbonate of ammonia is evolved.

g. Pickeringite and *bosjemanite* have been described under magnesia.

PHOSPHATES.

a. Turquois in the matrass decrepitates, yields some water, and turns black. B. B. infusible, but assumes a brown, glassy appearance and colours the flame green, partly from phosphoric acid and partly from a little oxide of copper. Dissolves easily in borax and S. Ph. to beads, yellowish-green when hot and pure green on cooling (copper and iron). The S. Ph. glass on coal with tin becomes opaque and red from suboxide of copper.

With soda swells at first and then is gradually dissolved to a semi-transparent glass, coloured with iron. With more soda becomes infusible, and with still more in a good R. F. some copper is reduced. The phosphoric acid can be detected by fusing the mineral with soda and silica, dissolving the mass in water and throwing down the phosphoric acid with acetate of lead (*vide* phosphoric acid). From the residue, insoluble in water, other constituents can be easily found out, after dissolving it in hydrochloric acid, as directed for lazulite (Magnesia).

b. Peganite from Langenstriegis occurs of an emerald-green, a greenish-gray, and a greenish-white colour.

The emerald-green variety yields water in the matrass and assumes an impure rose-colour. Ignited in a platinum crucible it loses 23·5 per cent. water. B. B. turns violet, cracks at a high heat, and is infusible, but assumes a glassy appearance and gives a greenish flame, partly owing to phosphoric acid and partly to a little oxide of copper. The latter causes a momentary azure-blue when the assay is previously moistened with hydrochloric acid. Dissolves easily in powder in borax and S. Ph.; the hot glass is yellow from iron, but nearly colourless on cooling. With cobalt solution the fine powder becomes blue.

The greenish-gray peganite behaves like the preceding, but in the matrass becomes reddish, in the crucible loses 24·1 per cent., and in the forceps becomes reddish-white. It seems also to contain somewhat more iron. The phosphoric acid in peganite can be detected in the same way as with turquois.

c. Fischerite behaves quite like the two preceding minerals.

d. Amblygonite in the matrass yields some moisture, which at a high temperature is acid and attacks the glass. B. B. fuses very easily to a clear bead and gives a yellowish-red flame of lithia and soda. The pulverized mineral moistened on platinum wire with sulphuric acid gives a momentary bluish-green flame of phosphoric acid. Dissolves largely and very easily in borax and S. Ph. to a clear, colourless glass. With fused S. Ph. in an open tube yields hydrofluoric acid (*vide* fluorine).

e. Wavellite in the matrass yields water, the last drops of which have an acid reaction on Brazilwood-paper. The glass is also attacked by the liberated hydrofluoric acid. Ignited in the covered platinum spoon it loses twenty-seven to twenty-eight per cent. of its weight.

B. B. swells and frequently is divided into fine acicular particles, which radiate from one point and are quite infusible, but turn white, if the mineral was not already white, and produce a bluish-green, phosphoric acid flame, most distinct after moistening with sulphuric acid. With the fluxes and cobalt solution like alumina, but the strongly saturated borax bead sometimes shows a little iron, while a manganese reaction is frequently obtained with soda and nitre. Is perfectly soluble in hydrochloric acid if free from silica.

f. Striegisan behaves in the matrass like wavellite, but its gray colour becomes somewhat darker. Ignited in the platinum spoon it loses 25·7 per cent. in weight. B. B. becomes grayish-white, is infusible, and gives a bluish-green flame, With the fluxes, etc., like wavellite, but shows rather more iron, and with S. Ph. a little silica separates. Leaves in hydrochloric acid only a trifling gray residue. Treated in the wet way like turquois, a little lime is found in addition to alumina and sesquioxide of iron.

g. *Variscite* yields in the matrass considerable water, having an alkaline reaction, and does not decrepitate, but assumes a feeble rose colour. Powdered and heated in a matrass with soda, carbonate of ammonia is evolved. B. B. quite infusible, but turns white and is unaltered in the R. F. The flame is tinged bluish-green. Dissolves rather easily in borax and S. Ph., even in fragments, to a clear, pale yellowish-green glass. With cobalt solution becomes blue. Fused on coal with soda, the fused mass decomposed by hydrochloric acid, and the solution evaporated to dryness, a mass is obtained which dissolves perfectly in water; it contains therefore no silica. If another portion is fused on coal with soda and silica, as described above for turquois, and further treated in the wet way, in addition to alumina trifling quantities of sesquioxide of iron and chromium and of magnesia can be found and the phosphoric acid detected with certainty.

MELLATE OF ALUMINA.

Mellite yields water in the matrass, and when heated to redness chars and emits a burnt odour. In the forceps and on coal burns white and then behaves like pure alumina with the fluxes, etc.

SILICATES.

The silicates under *α* and *β*, and the alumina-bearing silicates enumerated under the alkalies and preceding earths, after being tested for water in the matrass and as to fusibility, differ considerably in their behaviour; the following remarks, however, apply generally to silicates rich in alumina.

They dissolve with difficulty in borax to a clear glass, more or less yellow when iron is present.

With S. Ph. they are slowly decomposed and generally only when powdered, the bases dissolving and the silica remaining behind. When alkaline bases are present the bead opalesces on cooling. With a little soda they generally fuse to a bead, but with more soda give a slag-like mass, provided the bases are not combined with a high proportion of silicic acid.

Cobalt solution can only be employed when the silicate is infusible and contains little or no colouring metallic oxides and also not much magnesia. An infusible silicate of alumina free from these ingredients often assumes a fine blue colour when treated in fine powder with cobalt solution. When it is neither possible to determine the silicate with the blowpipe, nor to detect alumina, the method described for lime must be adopted, and also for rocks, dressed ore, and slags, which are to be examined for alumina.

The substance is decomposed by hydrochloric acid at once, or after fusion with soda and borax, then the silica and perhaps a trifle of baryta are separated, the protochloride of iron converted into sesquichloride of iron by nitric acid, and excess of ammonia gradually added to the acid solution, which contains the remaining bases. Alumina and sesquioxide of iron are thus thrown down together, with any chromium present, which appears as sesquioxide. When the substance contains much magnesia or protoxide of manganese, a very trifling quantity of these is also frequently present in the alumina and iron precipitate, which is filtered out, washed well with hot water, and heated while still moist with a solution of caustic potassa until the alumina is dissolved, leaving the dark-brown sesquioxide of iron either alone or mixed with the sesquioxide of chromium and exceedingly trifling quantities of magnesia and protoxide of manganese. The alkaline solution is diluted with water, the oxide of iron filtered out, and hydrochloric acid added to the filtrate until it reacts slightly acid, when the alumina is again precipitated with ammonia and may be collected on a filter, well washed with hot water, and tested with cobalt solution. Should glucina, which behaves similarly with potassa and ammonia, be suspected, the alumina may be tested for it, as will be described under glucina. If the tests with fluxes have shown chromium, the separated sesquioxide of iron should be examined for chromium (*vide* chromium). The method of detecting the other bases, lime, magnesia, and protoxide of manganese in the ammoniacal filtrate from the alumina, sesquioxide of iron, etc., is given under lime.

SILICATE OF ALUMINA WITH FLUORIDE OF ALUMINIUM.

Topaz (*pyrophysalite* and *pycnite*) are unaltered in the matrass. In the open tube with fused S. Ph. they yield hydrofluoric acid (*vide* fluorine). B. B. infusible, but the yellow topaz sometimes assumes a feeble rose colour. Dissolves slowly in borax to a clear glass, slightly yellow if iron is present. By S. Ph. they are slowly decomposed, the silica forming a skeleton, while the bead opalesces on cooling.

Finely powdered they become blue with cobalt solution.

According to Turner certain topazes fused on platinum wire with fluorite and bisulphate of potassa give a boracic acid flame.

ALUMINATES.

The behaviour of some of the above-named aluminates has been already stated under magnesia; that of the others will be described under glucina and zinc.

10. GLUCINA, BE.

Its occurrence in the mineral kingdom.

Glucina is not of frequent occurrence, being found only in combination with silica and alumina.

Examination for Glucina.

SILICATES.

Phenacite yields nothing volatile in the matrass. B. B. is infusible. Is dissolved with extreme difficulty by borax unless in fine powder, when it dissolves rather easily to a clear glass. In the strongly saturated glass white flocks are produced by flaming. The powder dissolves in S. Ph. and leaves a silica skeleton. With a little soda a milk-white bead, with more it swells and forms an infusible white slag. Cobalt solution produces a dull blue colour.

After fusing the fine powder with soda and borax on coal, and treating the compound with hydrochloric acid until the silica is separated, the glucina may be readily separated and further tested. The acid solution is made slightly ammoniacal, when glucina and any traces of sesquioxide of iron are thrown down. They are collected on a filter, washed thoroughly, and heated while still moist with solution of potassa, until the glucina is redissolved, leaving the oxide of iron. According to Schaffgotsch particular care must be taken that the potassa solution does not boil, otherwise some of the glucina may remain undissolved. After diluting the solution with water, filtering it, and making it slightly acid with hydrochloric acid, the glucina can be again thrown down by ammonia and may then be tested for alumina. To this end it is filtered out, thoroughly washed, and then shaken in a test tube with a large quantity of carbonate of ammonia solution, which dissolves the glucina and leaves the alumina. The latter, if found, may be collected, washed, and tested with cobalt solution. The ammoniacal solution of glucina is heated to boiling in a porcelain vessel, when the glucina goes down as basic carbonate, and this can be converted into pure glucina by ignition in the platinum capsule. For its blowpipe characteristics, see tables.

Euclase is unaltered in the matrass and its water cannot be detected here, being only expelled at a very high temperature. B. B. swells up into cauliflower-like points, whitens and fuses with difficulty on the edge to a white enamel. Dissolves slowly in borax to a clear, colourless glass, which cannot be flamed opaque. If a fragment is employed it first swells with slight effervescence and then whitens. Decomposed by S. Ph. with slight effervescence, leaving a white silica skeleton, while the glass remains clear and colourless, but opalesces on cooling. A reduction assay with soda on coal yields traces of tin.

Beryl is unchanged in the matrass. B. B. thin splinters become rounded and form a vesicular, colourless scoria; transparent varieties become milk-white. Dissolves in borax to a clear glass, fine green when cold if chromium is present. By S. Ph. it is very imperfectly decomposed; the assay remains nearly unaltered, but diminishes somewhat, showing that some silica is dissolved as well as the bases. The cold glass opalesces and is green if chromium is present. It is dissolved by soda to a clear, colourless glass, and according to Berzelius the yellow mineral from Broddbo and Finbo yields visible traces of tin by the reduction essay.

The method above given for phenacite is also employed in separating the glucina from euclase and beryl. The precipitate obtained by ammonia, containing the alumina, glucina, and sesquioxides of iron and chromium, is treated as before with potassa solution, which leaves the oxides of iron and chromium. These are collected on a filter, washed, and tested. The alumina and glucina are separated as before.

Helvite yields a little water in the matrass, otherwise unchanged. B. B. intumesces strongly and fuses with difficulty to a dark-yellow to brown bead, not free from bubbles. Dissolves slowly in borax to a clear violet glass, nearly colourless in the R. F. It is rather easily decomposed by S. Ph., yielding a silica skeleton and a colourless glass, opalescent on cooling. Swells at first with soda, then fuses to a black bead, chestnut-brown in the R. F. With more soda spreads out, sinks partly into the coal, and yields a sulphur reaction. With soda and nitre a strong manganese reaction.

To detect the glucina the finely-powdered mineral is dissolved in hydrochloric acid (paper moistened with acetate of lead shows that sulphuretted hydrogen is evolved), carefully evaporated to dryness, moistened with hydrochloric acid, dissolved in boiling-hot water, and the silica filtered out. The filtrate is further treated as directed for phenacite. The protoxide of magnesia can be precipitated in the ammoniacal filtrate from the glucina and sesquioxide of iron, either with sulphide of ammonium or a solution of S. Ph., and tested.

Danalite; B. B. fuses readily on the edges to a black enamel. With soda on charcoal gives a slight coating of oxide of zinc (and a sulphur reaction on silver foil). Perfectly decomposed by hydrochloric acid, with evolution of sulphuretted hydrogen and separation of gelatinous silica. (Dana.)

Leucophanite fuses, according to Erdmann, to a clear bead inclining to violet, which can be flamed opaque. According to Dana it whitens and shows purple phosphorescence in the matrass; in the forceps phosphoresces and fuses with intumescence to a clear, colourless glass, also produces an intense soda flame. Dissolves easily in borax to a clear amethyst-red bead. Is easily decomposed by S. Ph. and leaves a silica skeleton. In the open tube with fused S. Ph. shows fluorine. The lime and glucina may be easily found by the method given for phenacite; the ammoniacal filtrate from the glucina contains the lime, which is thrown down with oxalic acid.

COMBINATION OF GLUCINA AND ALUMINA.

Chrysoberyl is unaltered in the matrass. B. B. infusible, but heated in fine powder on coal becomes glassy on the edges. Dissolves slowly in borax and S. Ph. to a clear glass; in S. Ph. dissolves very slowly unless powdered. With soda only rendered dull on the surface; otherwise not attacked. The powder becomes blue with cobalt solution. The glucina is separated as described above for phrenacite, etc.

11. YTTRIA, $\dot{Y}$, AND ERBIA, $\dot{E}$.

Their occurrence in the mineral kingdom.

Yttria is but rarely met with and nearly always in common with oxide of erbium in various combinations.

a. As *fluoride of yttrium;*
b. With *phosphoric acid;*
c. With *tantalic acid;*
d. With *niobic acid;*
e. With *niobic* and *titanic acids;*
f. With *titanic acid;*
g. With *silicic acid* in a few silicates, some of which contain a little *water;*
h. With *titanic acid* and *combinations of silicic acid;*
i. With *carbonic acid.*

Examination for Yttria and Erbia.

Mosander, who discovered oxide of erbium in yttria, separated by chemical processes from various minerals, found that the accurate separation of these bases from each other was attended with numerous difficulties and could only be accomplished by using a considerable amount of material, while even then the bases could not always be isolated in a chemically pure state. Since blowpipe examinations are only made upon small quantities of a substance, we cannot expect to make any accurate separation of these bases until more certain and simple methods become known, and generally, therefore, the blowpipe examinations will be limited to the separation of both bases together, being only further extended when the substance to be examined is rich in yttria, or a larger amount of it is used than in the ordinary blowpipe analyses.

When not too small a quantity of yttria has been prepared, free from iron, uranium, and other easily separable metals, the oxides of which appear coloured after ignition, by the methods to be hereafter given, pure yttria may be obtained from it in the following way:

1. The separated yttria is dissolved in hydrochloric acid and dilute ammonia added to the solution in small quantities. The precipitate which forms after each addition, is filtered off, washed, and dried. By this means basic salts are obtained, the latter portions of which are perfectly colourless and contain only yttria. The preceding ones are reddish and contain constantly increasing quantities of erbia, which is especially found in the precipitates first formed.

2. The yttria separated from the minerals may be dissolved in nitric acid and the solution treated as above. When the precipitates are separately treated the first one yields a dark-yellow oxide, and the succeeding ones appear paler and paler, until the last, which is quite white and consists of pure yttria.

3. A similar separation may be accomplished by treating an acid solution with binoxalate of potassa, but certain precautions must be observed, and as the method is complicated and requires time, it will not now be further described.

FLUORIDE OF CALCIUM WITH FLUORIDES OF YTTRIUM AND CERIUM IN VARYING PROPORTIONS.

Yttrocerite from Finbo yields some water, which has a burnt odour. The dark variety becomes white in the matrass. On coal it is infusible, but upon addition of gypsum fuses to an opaque bead. With borax and S. Ph. a yellow bead while hot; this can be flamed opaque at a certain degree of saturation. With a little soda fuses to a bead, with more becomes less fusible, and when still more is added the soda sinks into the coal, leaving an infusible mass behind. (Berzelius.)

Yttrocerite from Broddbo decrepitates slightly in the matrass. B. B. infusible, but becomes milk-white, then brick-red, although not always similarly coloured. With gypsum it is infusible, owing to the large amount of fluoride of cerium. With the glass fluxes almost like sesquioxide of cerium. All yttrocerite yields hydrofluoric acid when tested in the open tube with fused S. Ph. (*vide* fluorine).

The yttria in yttrocerite can only be detected by the wet process. A little of the very fine powder is digested with sulphuric acid in a platinum dish, stirred up with a platinum wire and heated over the spirit-lamp under a chimney, until all of the fluorine and then all of the superfluous sulphuric acid are expelled, leaving sulphates behind. These are dissolved in dilute hydrochloric acid, water is added, and the diluted solution filtered, if any sulphate of lime should remain undissolved. From the clear solution protoxide of cerium and oxides of lanthanum, didymium, yttrium, and erbium are precipitated by adding ammonia in slight excess, and the precipitate is filtered out. From the ammoniacal filtrate lime is thrown down by oxalic acid. The precipitate formed by adding ammonia is washed with hot water, until the wash water is no longer clouded by oxalic acid. (If it were necessary to examine this precipitate for alumina or glucina it would require to be heated moderately with a solution of potassa, and if it contained sesquioxide of iron this would be afterward removed by a dilute solution of oxalic acid (*vide* phosphate of yttria); but, according to Berzelius, yttrocerite contains none of these.) The well-washed precipitate is then transferred to a small beaker glass, dissolved in a little hydrochloric acid, and diluted with water. In this solution is placed a crust of crystallized sulphate of potassa, so that it reaches above the surface of the liquid, and the whole is set aside for twenty-four hours, or else the solution, if not too dilute, is treated with a quite concentrated solution of sulphate of potassa, prepared with the aid of heat, and the whole is allowed to cool. In either case the result is a liquid saturated with sulphate of potassa, in which yttria and protoxide of cerium form double salts with the potassa and sulphuric acid. The yttria salt is soluble in the saturated solution of sulphate of potassa, while that of the protoxide of cerium is insoluble and falls to the bottom as a white powder. It is filtered out, washed with a saturated solution of sulphate of potassa, and dissolved in boiling-hot water, after which the protoxide of cerium is thrown down with potassa solution by the aid of heat, filtered out, washed thoroughly, and then ignited in the platinum spoon. During ignition it oxidizes to sesquioxide and assumes, if pure, a lemon-yellow colour; but if it contains didymium it assumes a cinnamon-brown hue. Oxide of lanthanum is white and therefore any admixture of it would not be perceptible, but upon treating the ignited oxide with nitric acid, evaporating it to dryness, and igniting the dry residue with access of air, the oxide of lanthanum may be extracted with very dilute nitric acid and precipitated with solution of potassa (*vide* cerium, lanthanum, etc.)

The yttria remaining in the solution is likewise precipitated by potassa with the aid of heat, filtered out, and ignited. To test it for erbia it is dissolved in hydrochloric or nitric acid and ammonia added in small portions. If the first precipitates appear yellow after ignition it may be assumed that the yttria contained erbia, since this has a dark yellow colour after ignition, while yttria is then pure white.

PHOSPHATE.

Xenotime intumesces slightly and fuses with difficulty on the edges, colouring the flame distinctly bluish-green after being moistened with sulphuric acid. It dissolves slowly in borax to a clear glass, slightly yellow from iron while warm, and otherwise like yttria. In S. Ph. it dissolves very slowly (distinction from apatite). The glass is colourless. With soda it is decomposed with effervescence to a light gray, infusible slag. Phosphide of iron is obtained by the test with metallic iron and boracic acid. (See Phosphorus.)

To detect the yttria with certainty, the very finely-powdered mineral is mixed with four to five times its weight of soda and fused, either in separate portions on platinum wire, or in the platinum spoon, until it no longer effervesces. The fused mass is covered with water in a small porcelain vessel and heated to boiling over the lamp. Phosphate of soda and the excess of carbonate of soda dissolve, leaving the insoluble yttria and some sesquioxide of iron, which is present in the mineral as basic phosphate. After filtering out the residue and washing it thoroughly, phosphoric acid may be very readily detected by testing a little of the filtrate (*vide* phosphoric acid).

The residue of yttria and sesquioxide of iron may be dissolved while still moist in hydrochloric acid, diluted, and the bases thrown down as hydrates by means of ammonia; they are thoroughly washed, transferred to a test tube, covered with a dilute solution of oxalic acid, and heated nearly to boiling over the spirit-lamp. Both vases are converted into oxalates, and the oxalate of sesquioxide of iron being soluble can be readily separated by filtration from the insoluble oxalate of yttria, which appears as a heavy, white powder. This being also insoluble in pure water is washed thoroughly, dried, and ignited. A special test is necessary to determine whether the yttria is pure, or contains erbia, and for this the quantity of yttria must not be too small. The sesquioxide of iron in the filtrate is separated by means of potassa, after some nitric acid has been added and the whole warmed. It is filtered out, washed, and tested B. B. with borax, if necessary.

TANTALATES.

Yttrotantalite, yellow and black. According to Berzelius they behave as follows:

Alone in the matrass they yield water, and the dark varieties turn yellow. Strongly ignited they become white, and the glass is attacked, while the expelled water turns Brazil-wood paper yellow at first and then bleaches it. B. B. infusible. Dissolve in borax to a nearly colourless glass, becoming opaque of itself with a large addition. Dissolve in S. Ph., leaving a white skeleton of tantalic acid, which on continued blowing also dissolves.

The *black* variety from Ytterby gives a glass which assumes on cooling a feeble rose colour, after treatment in R. F., owing to the presence of tungsten.

The *yellow* variety from Ytterby affords a faint but fine green bead, due to uranium. *Yttrotantalite* from Finbo and Kararfvet affords a strong iron colour, which obscures the uranium reaction. Soda decomposes, but does not dissolve them. On platinum foil they show manganese, and by reduction with borax and soda yield traces of tin. The mineral from Finbo, however, contains so much iron that the tin cannot be thus detected.

Hielmite decrepitates in the matrass, crumbles and yields water. In O. F. infusible, but becomes brown. Dissolves readily in borax to a clear glass, cloudy by flaming. With S. Ph. a bluish-green glass. With soda on charcoal gives metallic spangles. Dana states that the borax bead remains unchanged on flaming.

NIOBATES.

Fergusonite yields a little water in the matrass. B. B. on charcoal becomes first dark, then pale-yellow, but is infusible. Dissolves with difficulty in borax to a glass which is yellow while hot, and if saturated, can be made cloudy and dirty yellowish-red by flaming; the undissolved portion is white. Dissolves slowly in S. Ph. to a glass, yellow in O. F., colourless in R. F., or inclining to red if well saturated; it then is readily made cloudy by flaming, or on cooling, but this is not the case with a moderate addition of the mineral. The portion remaining undissolved is white.

Fused with tin the S. Ph. glass remains colourless, but the undissolved, white portion of the assay acquires a flesh-red shade. It is decomposed by soda without being dissolved, and leaves a reddish slag. Reduced with enough soda it affords some metallic tin. (Berzelius.)

Samarskite, according to G. Rose, behaves as follows:

In the matrass decrepitates somewhat, glows, cracks open, and blackens. B. B. fuses on the edges to a black glass. With borax in O. F. a yellowish-green to reddish glass; in R. F. a yellow to greenish-black glass, becoming opaque and yellowish-brown by flaming. With S. Ph. in O. F. a clear, emerald-green glass, unaltered in R. F. With soda on platinum foil a manganese reaction. Fused with bisulphate of potassa forms a fluid, red mass, which is yellow when cold.

Tyrite decrepitates strongly, yields water, and becomes yellow. With borax yields a glass which is reddish-yellow while hot, and colourless when cold. With S. Ph. affords a silica skeleton, and the glass is greenish-yellow while hot, green on cooling.

According to Dana, *samarskite* in powder is sufficiently decomposed on boiling with sulphuric acid to give a blue colour when the acid fluid is treated with metallic zinc or tin; while *fergusonite* evaporated with sulphuric acid yields a white residue, which treated with hydrochloric acid and metallic zinc, gives a bluish-green colour.

COMBINATIONS WITH TITANIC AND NIOBIC ACIDS.

Euxenite is infusible. With borax and S. Ph. dissolves to glasses, yellow while hot, but the S. Ph. glass, not too slightly saturated, becomes yellowish-green on cooling, owing to uranium. (Scheerer.) Fused with caustic potassa, the mass dissolved in water, the solution neutralized with hydrochloric acid, and the resulting precipitate boiled with concentrated hydrochloric acid and tinfoil, a clear sapphire-blue fluid results, which changes to olive-green and finally bleaches. The mineral is sufficiently attacked on evaporation with sulphuric acid to give a whitish residue, which affords the characteristic blue reduction test, when treated with metallic zinc or tin. (Dana.)

Polycrase decrepitates in the matrass and gives traces of water. In the forceps glows and assumes a light grayish-brown colour. Dissolves in borax in O. F. to a clear yellow glass, which assumes a brown colour in R. F., especially in adding tin. With S. Ph. likewise dissolves to a clear yellow to yellowish-brown glass, passing into greenish on cooling. In R. F. the colour becomes darker. (Scheerer.) Decomposed by evaporation with concentrated sulphuric acid, the product treated with hydrochloric acid and boiled with metallic tin or zinc gives a deep azure-blue colour, which does not fade. (Dana.)

Æschynite yields some water and traces of hydrofluoric acid in the matrass. B. B. swells and changes from a black to a rusty-brown colour. Is rather easily dissolved by borax in O. F. to a clear glass, yellow while hot, colourless on cooling. Treated with tin in R. F. this glass becomes blood-red. It dissolves less easily in S. Ph.; a small addition affords a clear, colourless glass, which is readily made cloudy by separation of some white substance, when more of the assay is added. With tin in R. F. the glass assumes an amethyst colour. The mineral effervesces with soda, but is not dissolved and yields nothing metallic. (Berzelius and Herrman.) It is sufficiently decomposed by sulphuric acid to show the reduction test with zinc. (Dana.) The wet process must be partly employed in order to detect with certainty the separate constituents of the foregoing tantalates, niobates, and titanates, which may contain, in addition to the corresponding acids, tungstic acid, and as bases, lime, yttria, zirconia, and oxides of cerium, lanthanum, iron, manganese, uranium, and tin.

The following method is employed:

1. A sufficient amount of the very fine powder is fused in the platinum spoon with eight parts by weight of bisulphate of potassa in several portions, each portion being treated until everything is melted to a clear fluid, when the mass is at once poured out upon the steel anvil. When all has been thus fused, the mass is crushed in the steel mortar and then pulverized as fine as possible in the agate mortar. The powder is warmed with sufficient water, not being allowed to boil, and the following ingredients then dissolve:

$$\dot{\mathrm{Ca}},\ \dot{\mathrm{Y}},\ \dot{\mathrm{Ce}},\ \dot{\mathrm{La}},\ \dddot{\rlap{-}\mathrm{F}\mathrm{e}},\ \dot{\mathrm{Mn}},\ \dddot{\rlap{-}\mathrm{U}};$$

while there remain as a white residue:

$$\overset{\cdots}{\ddot{\mathrm{Ta}}},\ \ddot{\mathrm{Nb}},\ \dddot{\mathrm{W}},\ \ddot{\mathrm{Sn}}.$$

If the mineral contains $\ddot{\mathrm{Ti}}$, $\ddot{\mathrm{Zr}}$, and $\ddot{\mathrm{Th}}$, a portion of these is also likely to remain undissolved.

After filtration the residue is washed with hot water, to which a few drops of hydrochloric acid may well be added to secure the perfect separation of the sulphate of lime and sesquioxide of iron, and it may then be examined for all the $\ddot{\mathrm{Nb}}$, $\dddot{\mathrm{Ta}}$, and $\dddot{\mathrm{W}}$, as well as a portion of the $\ddot{\mathrm{Ti}}$, $\ddot{\mathrm{Zr}}$, and $\ddot{\mathrm{Th}}$, together with the trifling quantity of $\ddot{\mathrm{Sn}}$ sometimes present.

2. The residue is dried, finely pulverized, mixed with five volumes of carbonate of potassa, moistened with water, and melted in separate portions on stout platinum wire, until all is in quiet fusion, the melted mass being shaken off after each fusion. It is then pulverized and treated in a porcelain dish with cold water, to dissolve the excess of carbonate of potassa, the solution is drawn off with a pipette, and the residue treated with fresh water, which is heated to boiling. This dissolves $\dddot{\mathrm{Ta}}$, $\ddot{\mathrm{Nb}}$, $\dddot{\mathrm{W}}$, $\ddot{\mathrm{Th}}$, and most of the $\ddot{\mathrm{Sn}}$ combined with potassa, leaving the greater part of the $\ddot{\mathrm{Ti}}$ and all of the $\ddot{\mathrm{Zr}}$ behind. The solution is filtered off, the residue well washed, and the solution added to the first, in case a portion of the newly-formed potassa salts was soluble even in cold water, the whole acidified with hydrochloric acid, evaporated to dryness at a moderate heat, and the dry mass treated with hot water, which dissolves the chloride of potassium and most of the bichloride of tin, leaving behind $\dddot{\mathrm{Ta}}$, $\ddot{\mathrm{Nb}}$, $\ddot{\mathrm{Th}}$, and $\dddot{\mathrm{W}}$, with a trace of $\ddot{\mathrm{Sn}}$. The solution, which may contain tin, is treated with a little sulphide of ammonium, shaken, and a few drops of dilute hydrochloric acid added to it, when a yellow precipitate of sulphide of tin will form if tin is present. In order to free the tantalic or niobic acid from tungstic acid and binoxide of tin it is necessary to treat the mixture directly upon the filter with sulphide of ammonium. The neck of

the funnel is closed with a cork, the still moist mixture covered with sulphide of ammonium, and a watch-glass placed over the funnel, when the whole is set aside in a warm place. After all of the tungstic acid and oxide of tin have been dissolved as sulphides the funnel is opened, the solution allowed to flow off, and the remaining acid thoroughly washed. If it is not white it should be at once covered with dilute hydrochloric acid, while on the filter, so as to dissolve any traces of sulphide of iron present. The way in which it is further tested will be given under the examination for tantalum and niobium. Any thoria present can be separated by means of oxalic acid.

The dissolved sulphide of tungsten, with the traces of sulphide of tin which may be present, is thrown down by hydrochloric or nitric acid, filtered out, and the precipitate, rich in sulphur, after being freed from the excess of sulphur on coal in O. F. and then thoroughly ignited, is tested with S. Ph. on platinum wire for tungstic acid, and with soda and borax on coal for tin.

The residue which remains after separating the Ta, Nb, and W, and may contain Ti, Zr, and traces of Sn, is first tested with S. Ph. on platinum wire for titanic acid; it is then tested with soda on platinum foil, and if during the fusion the fluid mass is clear where most strongly heated, the residue consists only of titanic acid, but if not, since zirconia does not melt to a clear fluid mass with soda, it consists of titanic acid and zirconia, provided the test with S. Ph. has shown the presence of titanic acid. A portion of the residue may also be tested for tin by a reduction assay.

3. To the solution of the sulphates, containing free hydrochloric acid, ammonia is added in slight excess and with constant stirring; by this means (assuming that the solution contains also Ti, Zr, and Th) are precipitated, Ti, Y, Th, Zr, Ce, La, Fe, and Ʉ, while Ca and Mn remain for the most part in solution. After filtering out the precipitate and washing it well with hot water, the lime is thrown down from the ammoniacal filtrate with oxalic acid and

any manganese present with S. Ph. or sulphide of ammonium.

4. The precipitate obtained by means of ammonia is washed, dried, and dissolved with the aid of heat in sulphuric acid, diluted with an equal amount of water. Water is added to the clear solution and the whole boiled, when any titanic acid present is thrown down, filtered out, and tested B. B.

5. The filtrate freed from titanic acid is neutralized with potassa until it has but a very feebly acid reaction, and is then saturated with a concentrated solution of neutral sulphate of potassa, freshly prepared with the aid of heat. The whole is then allowed to cool, during which time a precipitate, partly flocculent and partly pulverulent, generally settles, consisting of basic sulphate of zirconia and sulphates of the oxides of cerium and lanthanum with potassa. If the mineral contains thoria, it is also present in this precipitate, which is collected on a filter and washed with a saturated solution of sulphate of potassa. The filtrate contains all of the Y, Ꞙe, and Ʉ. Another vessel being placed below the funnel, the salts on the filter are washed with pure boiling-hot water, which dissolves the double salts of cerium and lanthanum, leaving the basic sulphate of zirconia and the sulphate of thoria. These two can only be separated conveniently in case a considerable quantity is treated, and then oxalic acid is used, in which the zirconia dissolves, while thoria is insoluble. The oxides of cerium and lanthanum are thrown down from their solution by potassa, and then separated by means of dilute nitric acid (*vide* cerium and lanthanum).

6. The solution, which may still contain Y, Ꞙe, and Ʉ, is treated with a slight excess of potassa, and all three bases thrown down as hydrates. The precipitate is transferred to a small filter, well washed with hot water, and then moderately heated in a small porcelain vessel with a dilute solution of oxalic acid, until the residue appears pure white. Ꞙe and Ʉ dissolve, while the oxalate of yttria remains, and after filtration is washed, dried, ignited, and tested according to p. 168.

After adding some nitric acid, the Ꞙe and Ʉ in the solution are again thrown down with potassa, the whole warmed, filtered, washed, and the precipitate, while still moist, treated with carbonate of ammonia, which dissolves the $\bar{\mathrm{U}}$ and leaves the $\mathrm{\bar{F}e}$. From the ammoniacal filtrate the $\bar{\mathrm{U}}$ can be thrown down by boiling the fluid for some time, or by gradually adding hydrochloric acid to feebly acid reaction and then adding ammonia. The oxides thus separated may then be very easily recognized B. B. with glass fluxes.

TITANATE.

Polymignite.—B. B. on coal is unchanged. Dissolves easily in borax, the glass showing iron and, when much is added, becoming opaque and usually brownish-yellow by flaming. With still more it becomes opaque on cooling. With tin the bead assumes a red colour, inclining to yellow.

In S. Ph. dissolves with difficulty to a glass, which is reddish in the R. F. and also when treated with tin.

It is decomposed by soda and assumes a grayish-red colour, but cannot be fused. On platinum foil a manganese reaction. (Berzelius.)

The separate ingredients are found in the wet way, as described for tantalates, niobates, and titanates.

SILICATES.

a. Gadolinite from Ytterby, Finbo, and Broddbo.

With regard to these gadolinites and their behaviour Berzelius makes the following observations :—

They are of two kinds; one (α) as vitreous as if it were a fragment of black glass; the other (β) splintery in its fracture and not so largely conchoidal. It is apparently an intimate mixture of gadolinite and orthite.

Var. α. If heated in a matrass, nearly to the melting point of the glass, the assay glows quickly, as if on fire; it also swells somewhat, becomes light grayish-green, and cracks here and there if large. On coal the same; it is infusible, but the thin edges become black in a strong flame.

Var. β. Alone it swells up into cauliflower-like ramifications and becomes white, but seldom glows like variety α.

Both varieties behave similarly with the glass fluxes, dissolving readily in borax and yielding a strong iron reaction. S. Ph. dissolves them with great difficulty, the glass showing iron, and the fragment becoming rounded, but remaining white and opaque, so that the silica is not separated (chief distinction from gadolinite of Kararfvet). With soda they are dissolved to a reddish-brown, half-fused slag, but variety β fuses to a globule, when there is not too much soda. No manganese reaction can be obtained.

According to Damour and Descloizeaux different varieties of gadolinite from Ytterby show the following behaviour B. B. Some yield water, glow more or less brightly, swell, and are infusible, excepting one variety, which yields a black slag. The dark colour of them ineral is changed, becoming greenish-gray to grayish-white. G. from Broddbo and Finbo is similar; G. from Fahlun yields water, shows no perceptible glowing, becomes dark brown in the forceps, and is infusible.

b. Gadolinite from Kararfvet. In the matrass yields a little water; on coal whitens, and in a strong flame fuses to a dark pearl-gray or reddish, opaque glass, without swelling. Dissolves readily in borax to a clear glass, showing little iron. If saturated, the opaque glass crystallizes on cooling and becomes gray, inclining to red or green, but the enamel-like opacity, afforded by yttria alone, cannot be produced.

Dissolves in S. Ph., leaving a silica skeleton and forming a nearly colourless glass, opalescent on cooling. With soda fuses with difficulty to a grayish-red slag, and on platinum foil gives a manganese reaction (Berzelius). According to Damour and Descloizeaux it glows brightly B. B., swells a little, fuses with difficulty on the edges, and becomes gray.

c. Gadolinite from Hitteroe has been chemically examined by Scheerer. A sufficiently large fragment heated to low redness in a partially closed platinum spoon, is observed to glow very strongly, the light spreading from one point throughout the whole. B. B. infusible; with the fluxes shows iron and silica.

According to Damour and Descloizeaux it glows when heated to redness, cracks, remains transparent, and is infusible.

d. Alvite is infusible. With borax a greenish-yellow glass, colourless on cooling. With S. Ph. a yellow glass, becoming green and finally colourless on cooling, and showing no titanium reaction with tin.

To detect yttria and the other ingredients, not to be recognized by the blowpipe, the foregoing silicates must be examined with the aid of the wet process. If not previously ignited, they can be completely decomposed by hydrochloric acid, and the following method may therefore be adopted:—

1. The very fine powder is heated with aqua regia, until completely decomposed; the whole is then gently evaporated to dryness, moistened with hydrochloric acid, the resulting salts dissolved in hot water, and the separated silica filtered out; after which it is well washed and can be tested B. B.

The solution may contain the following bases:

$\dot{\mathrm{C}}\mathrm{a}$, $\dot{\mathrm{M}}\mathrm{g}$, $\ddot{\bar{\mathrm{A}}}\mathrm{l}$, Be, Y, Ce, $\dot{\mathrm{L}}\mathrm{a}$, $\bar{\mathrm{F}}\mathrm{e}$, $\dot{\mathrm{M}}\mathrm{n}$, with traces of $\dot{\mathrm{K}}$ and $\dot{\mathrm{N}}\mathrm{a}$.

2. To separate these, excess of ammonia is gradually added to the acid solution, and $\bar{\mathrm{A}}\mathrm{l}$, Be, $\dot{\mathrm{Y}}$, $\dot{\mathrm{C}}\mathrm{e}$, La, and $\bar{\mathrm{F}}\mathrm{e}$ are thrown down, leaving $\dot{\mathrm{C}}\mathrm{a}$, $\dot{\mathrm{M}}\mathrm{g}$, and Mn dissolved. The precipitate is collected on a filter, well washed, and the bases in the ammoniacal filtrate are separated by means of oxalic acid and S. Ph.

3. The precipitate formed by ammonia generally contains but little alumina and glucina and is dissolved in a small porcelain dish with just the necessary amount of dilute hydrochloric acid, the solution constantly stirred and excess of potassa added, after which the whole is heated, but not to boiling. At first all the bases are thrown down by the potassa, but $\dot{\bar{\mathrm{A}}}\mathrm{l}$ and Be redissolve, especially when the solution is heated. After diluting the whole with water, filtering and washing the precipitate thoroughly with hot water, the earths in the alkaline filtrate are separated as directed under glucina.

4. The moist precipitate, containing $\dot{\mathrm{Y}}$, $\dot{\mathrm{C}}\mathrm{e}$, $\dot{\mathrm{L}}\mathrm{a}$, and $\ddot{\bar{\mathrm{F}}}\mathrm{e}$, is heated with dilute oxalic acid, which removes the $\dot{\bar{\mathrm{F}}}\mathrm{e}$. The residue of oxalates is filtered out, washed, dried, and ignited with access of air; the resulting oxides are then dissolved in dilute hydrochloric acid and separated by means of sulphate of potassa, as already described under yttrocerite. The sesqui-

oxide of iron in the solution is thrown down by potassa, after some nitric acid has been added, and is tested B. B.

SILICATES CONTAINING TITANIC ACID.

For *Keilhauite*, which belongs here, see *Lime*.

12. ZIRCONIA, Zr.

Its occurrence in the mineral kingdom.

Zirconia is of rare occurrence, and is always found in combination with bases and acids.

Examination for Zirconia.

Wöhlerite behaves, according to Scheerer, as follows:—

Strongly heated fuses quietly to a yellowish glass. With borax, S. Ph., and soda shows reactions for manganese, iron, silica, and gives traces of tin.

To detect the zirconia and hyponiobic acid with certainty, the very fine powder is heated with concentrated hydrochloric acid, until fully decomposed. According to Scheerer, this dissolves Na, Ca, Mg, Zr, $\overline{\mathrm{Fe}}$, and Mn, leaving Si and $\overline{\mathrm{Nb}}$. The residue, collected on a filter, well washed, and dried, is mixed and fused with five volumes of carbonate of potassa, being moistened and melted on platinum wire by separate portions, so that after each addition the melted mass is clear and fluid. The beads shaken off from the wire, which become opaque on cooling, are pulverized and treated first with cold water in a porcelain vessel to dissolve out the carbonate and silicate of potassa, and after the insoluble salt has settled and the supernatant liquid has been removed with a pipette, the residue is dissolved in boiling water, the solution added to the first solution, and the whole tested for the acids in question, as will be directed under tantalum and niobium; or else the solutions may be separately tested.

The acid solution of the bases is treated with a slight excess of ammonia, which precipitates Zr, $\overline{\mathrm{Fe}}$, and a little $\dot{\mathrm{C}}\mathrm{a}$. The precipitate is filtered off, washed with cold water, and warmed with dilute oxalic acid, when Zr and $\overline{\mathrm{Fe}}$ dissolve and leave the lime as oxalate. The latter is filtered out and some nitric acid added to the filtrate, after which $\dot{\mathrm{Zr}}$ and $\mathrm{\bar{F}e}$ are thrown down by potassa, collected on a filter, washed with hot water, dried, and gently ignited in the platinum spoon. It must then be triturated in the mortar, and is afterward digested with hydrochloric acid, which removes $\mathrm{\bar{F}e}$ and leave the zirconia almost pure white. This can be now tested B. B., while the oxide of iron may be likewise tested with borax, after precipitating it with ammonia. If the Zr and $\dot{\mathrm{\bar{F}e}}$ precipitate formed by potassa is digested with sulphide of ammonium, the iron is converted into sulphide, and when the liquid is decanted from it, after a time, and the black residue mixed with an aqueous solution of sulphurous acid, the sulphide of iron is dissolved, leaving the zirconia nearly colourless.

Of the other bases still dissolved in the ammoniacal solution, viz., Na, Ca, Mg, and Mn, the $\dot{\mathrm{C}}\mathrm{a}$ is thrown down with oxalic acid, and the Mg, and Mn with S. Ph., as directed under lime and magnesia. The soda is indicated by testing the mineral in the forceps.

Eudialyte yields a little water in the matrass. B. B. fuses easily to a greenish-gray bead; according to Damour, to a translucent, dark green glass, and gives an intense reddish-yellow flame. With borax is easily dissolved to a clear glass which shows a little iron and cannot be flamed opaque. Is easily decomposed by S. Ph., the silica skeleton swelling so as to alter the round shape of the bead. According to Berzelius, this forms a distinction between *eudialyte* and the otherwise similarly acting *garnets*. With little soda a difficultly fusible glass; with more goes into the coal. On platinum foil a manganese reaction. With a bead of S. Ph. containing oxide of copper, a chloride of copper flame (*vide* chlorine).

Gelatinizes with hydrochloric acid; the acid solution diluted and boiled with tin, to reduce the iron to protochloride, imparts a deep orange to turmeric-paper.

To detect the zirconia and lime the fine powder is dissolved in hydrochloric acid, tantalic acid and silica separating, evaporated carefully until nearly dry, dissolved in water, nitric acid added to form sesquichloride of iron, and the solution filtered.

glass. With S. Ph. and tin on coal a titanic acid reaction is produced. Is not dissolved by soda, but with the reduction assay traces of tin can be found. (Berzelius.)

The zirconia can be detected by fusing the fine powder with eight times its weight of bisulphate of potassa in the platinum spoon, treating the powdered mass with water, filtering out the silica, and then diluting with much water. A few drops of nitric acid are now added and the titanic acid thrown down as thoroughly as possible by continued boiling. After settling, this is filtered out and the acid solution, containing Zr, Ca, Mg, and Fe, treated with ammonia, which perfectly separates the Zr with the Fe; a little Ca goes down with them, but nearly all the Mg remains in solution. The further treatment of these bodies is indicated in the directions given for the preceding minerals.

Zircon, according to Berzelius, behaves as follows:—

The colourless, transparent variety is unaltered; the clear, red (*hyacinth*) loses colour, becoming clear as water, or very slightly yellow. The opaque, brown variety becomes white and resembles cracked glass. The dark variety from Finbo yields some moisture, becomes milk-white, and then appears as if weathered. All are perfectly infusible.

In borax zircon dissolves with difficulty to a clear glass, with which it shows the same behaviour as zirconia. Not attacked by S. Ph., the glass remaining colourless in O. F. and R. F. Insoluble in soda, the edges only being slightly attacked and the soda going into the coal. On platinum foil most zircons show traces of manganese.

Auerbachite behaves like zircon.

Malacon, according to Scheerer, behaves as follows:

In the matrass yields some water. B. B. is infusible. The fine splinters become white and opaque in borax and S. Ph., but are not dissolved. The extremely fine powder dissolves slowly in borax, showing a little iron; it is also decomposed by S. Ph., leaving the silica alone. In small fragments it is not attacked by soda.

Catapleiite yields water in the matrass. B. B. fuses easily to a white enamel. In borax dissolves with difficulty to a clear, colourless glass.

Easily soluble in hydrochloric acid without gelatinizing; the dilute acid solution colours turmeric-paper orange-yellow.

Tachyaphaltite yields water containing fluorine. B. B. becomes white, but does not fuse.

Holmite (variety of *seybertite*), analyzed by Richardson, becomes colourless and opaque B. B. and gives a slight iron reaction with borax.

To detect zirconia in zircon and the following minerals, their very fine powder is fused with one and a half volumes of soda and three of borax on coal in the O. F., the clear bead pulverized, treated with hydrochloric acid, and very gently evaporated nearly to dryness, so as to separate the silica. Rapid evaporation at a high temperature would cause much zirconia to remain undissolved in the subsequent treatment. The nearly dry mass is treated with a sufficient quantity of water, and the silica filtered out, after which any protochloride of iron must be transformed into sesquichloride with nitric acid. The zirconia and sesquioxide of iron are then thrown down by ammonia, and the very voluminous precipitate concentrated, so that it may be filtered out more easily, by boiling the whole. The little yttria and lime in malacon are here to be disregarded. The separation of the sesquioxide of iron from the zirconia is effected as before in case of *wöhlerite* and *eudialyte.* The ammoniacal filtrate can be further tested with oxalic acid, salt of phosphorus, and sulphide of ammonium, for other constituents.

Thoria, said to be present in tachyaphaltite, would be found in the precipitate of sesquioxide of iron and zirconia, and would remain behind on digesting this in oxalic acid.

According to Richardson, *holmite* contains only 2·05 per cent. zirconia, which could not be detected with certainty in the small quantity here treated.

13. THORIA, Th.

Its occurrence in the mineral kingdom.

Thoria is of very rare occurrence; it has been found:

a. In a combination of *hyponiobic acid* or *niobic acid*, with several bases;

b. Combined with *silicic acid;*

c. With *phosphoric acid.*

Examination for Thoria.

SILICATE OF THORIA.

Thorite behaves, according to Berzelius, as follows:—

In the matrass yields water and turns brownish-red. B. B. on coal infusible. Dissolves easily in borax, and the saturated glass is opaque on cooling, but if otherwise cannot be made opaque by flaming. The glass shows iron. With S. Ph. it leaves a silica skeleton; the glass shows iron and opalesces in cooling. With soda on coal a yellowish-brown slag; on platinum foil manganese. By a reduction assay, minute malleable globules of tin and lead are obtained.

Orangite, according to Bergmann, behaves thus:—

In the platinum spoon small splinters generally crumble to a dark brown mass, and again assume their orange colour on cooling; the larger fragments become opaque. Heated in the alcohol flame in the forceps, fragments decrepitate slightly and particles which fly off glow brightly, without afterward showing change of colour. Infusible on coal, the edges only being sometimes slightly glazed, perhaps owing to foreign bodies. With soda only the silica is dissolved; the remaining substances can be detected as yellowish particles in the opaque glassy mass with the magnifier. With borax a yellowish bead, colourless when cold; but with S. Ph. in the O. F. a reddish glass, colourless on cooling, which becomes yellowish in the R. F. and is then also colourless when cold.

To detect the thoria in these minerals they are fused in fine powder with soda and borax on coal beside a silver button in the R. F., until any oxides of tin and lead are reduced and united with the silver and the fused glass appears quite clear. This is pulverized and treated as usual with hydrochloric acid, the dry salt obtained by evaporation is moistened with hydrochloric acid dissolved in water, and the silica filtered out. After boiling the solution with a few drops of nitric acid to peroxidize the iron, a slight excess of ammonia is added, which throws down thoria, sesquioxides of iron and uranium, and part of the protoxide of manganese. The amount of alumina present is not worthy of notice. The washed precipitate, while moist, is dissolved in dilute sulphuric acid, and the solution evaporated to a small volume. During the evaporation neutral sulphate of thoria separates as a white, loose mass, from which the acid solution of the other bases is decanted after some time. This salt is then washed with boiling water, dried, and converted into pure thoria by ignition.

To separate the thoria still contained in the wash-water and the solution of the other bases, the whole is evaporated to a rather small volume, neutralized with carbonate of soda, and then a boiling-hot saturated solution of sulphate of potassa is added. While cooling, a double sulphate of thoria and potassa separates, which is washed with a cold, saturated solution of sulphate of potassa, and then dissolved in boiling-hot water containing some sulphuric acid, after which the thoria is precipitated by ammonia, dried, and ignited. The oxides of iron and uranium are separated from the solution as directed under yttria. The lime is found in the first ammoniacal filtrate by adding oxalic acid.

2. Examinations for Metals or their Oxides.

I. CERIUM, Ce; LANTHANUM, La; DIDYMIUM, Di.

Occurrence of these metals in the mineral kingdom.

Cerium is one of the rare metals, and occurs in the following minerals, almost always in connection with more or less lanthanum and didymium.

a. Combined with *fluorine* as neutral fluoride;

b. Combined with *phosphoric acid;*

c. With *carbonic acid;*

d. With various *metallic acids,* as *niobic, hyponiobic,* and *titanic acids;*

e. With *silicic acid* in several silicates;

f. In *silicates* containing *titanic acid;*

g. With *phosphoric acid.*

Examination for Cerium, Lanthanum, and Didymium.

a. General examination for cerium, lanthanum, and didymium.

The great difficulty of completely separating the three oxides from one another renders it seldom possible to undertake this with the trifling quantity employed in blowpipe analysis. The oxide of cerium can be separated in approximate purity from the mixed oxides obtained, after they have been ignited, by first treating them with dilute and then with concentrated nitric acid, which extracts the lanthanum and didymium. Upon evaporating this solution, igniting the salt, and again treating the oxides with very dilute nitric acid, any oxide of cerium which had been dissolved remains now undissolved. From the solution of lanthanum and didymium the oxides are thrown down with ammonia and dissolved in sulphuric acid. The dry salt being then dissolved to saturation in water at 43° to 45° F. and the solution warmed to 100°, sulphate of lanthanum separates, leaving the didymium salt in the solution, from which it can be precipitated by potassa. The oxides may be obtained still purer by repeating this process. Another method, not so suitable for small quantities, consists in separating the oxides by means of hydrate of potassa and chlorine.

Pure sesquioxide of cerium is yellow, inclining to red; the impure oxide is brick-red. Proto-sesquioxide, obtained by igniting the oxalate of the protoxide in the air, is white, with a shade of yellow, and is orange-red when heated. Oxide of lanthanum is colourless; ignited oxide of didymium is white and the peroxide brown.

In many minerals, containing besides oxides of cerium, lanthanum, and didymium, no considerable quantity of other colouring metallic oxides, viz., *fluocerite*, the *phosphates* and *carbonates*, and *cerite*, the oxides mentioned can be found with comparative ease. With borax and S. Ph. in the O. F. red or dark yellow beads are obtained, according as more or less has been dissolved, and these lose their colour on cooling, as well as in the R. F., to such an extent that the S. Ph. bead becomes quite colourless. The borax bead can also be rendered opaque or enamel-white by flaming, and the more quickly the less silica there is in the mineral. In the other minerals, which contain also oxides of iron and uranium, or titanic acid, the cerium, etc., cannot always be detected with certainty except by the aid of the wet ways, as has been already described for many of the above minerals under yttria.

b. Behaviour of minerals before the blowpipe.

FLUORIDES.

Fluocerite in the matrass yields water and at the melting point of glass the matrass is attacked at a distance from the assay. The water colours Brazilwood-paper yellow; the assay piece becomes white. In the open tube, when the flame is directed within the tube, the interior is attacked; the condensed water turns Brazilwood-paper yellow, and the assay piece assumes a dark yellow colour. On coal infusible, but darkens in colour. With borax and S. Ph. reacts like oxide of cerium containing oxides of lanthanum and didymium. With soda it is disintegrated and swells, but is not dissolved; the soda goes into the coal, leaving a gray mass.

Fluocerine yields water and becomes darker (Berzelius). On coal changes colour, and when heated nearly to redness appears black, but on cooling becomes dark brown, then fine red, and finally dark yellow. This change of colour distinguishes it from fluocerite. It is infusible. With fluxes like fluocerite, but is not disintegrated by soda, except by a strong and long-continued blast.

PHOSPHATES.

Churchite.—B. B. in the tube yields acid water, becoming opaque. In O. F. becomes reddish. With borax in O. F. a bead, orange-yellow and opaline while hot, colourless or slightly amethystine when cold. (Dana.)

Cryptolite is unaltered by moderate heating. Soluble in concentrated sulphuric acid. *Phosphocerite,* according to Chapman, vitrifies partially on the edges, tinging the flame slightly green. Affords the reaction of phosphoric acid and also of cerium; but produces with borax and S. Ph. a glass, pale violet-blue when cold, owing either to didymium, or a minute portion of cobalt ore. (Dana.) Cryptolite remains behind, when the green and reddish opatites of Arendal are dissolved in acids, and is similarly found in the cobalt ores of Tunaberg.

Monazite.—B. B. becomes dark gray, and when strongly heated the crystal faces become lustrous. Moistened with sulphuric acid it gives a bluish-green flame. Dissolves in borax and S. Ph. to a glass, yellow when hot and nearly colourless when cold; the same in both flames; with borax in the R. F. the strongly saturated glass becomes enamel-white by flaming. With S. Ph. and tin on coal a slight titanium reaction. With soda on coal a little tin is obtained, and on platinum foil a manganese reaction. With boracic acid and iron phosphide of iron is formed. (Kersten.)

Monazitoid yields water. B. B. becomes strongly luminous, without fusing. With fluxes like monazite. (Hermann.)

CARBONATES.

Parisite in the matrass yields water and carbonic acid, becoming cinnamon-brown and friable. B. B. phosphoresces, but is infusible. With borax a yellow bead, colourless on cooling. (Bunsen.) With fused S. Ph. in the open tube gives the fluorine reaction. (Dana.)

Kischtimite yields water and becomes darker in the matrass. B. B. at a moderate heat becomes dull, opaline, and yellow; at a higher heat glows, and when cold has a high lustre and is brick-red. With borax in O. F. a glass, yellowish-red when hot, feebly yellowish on cooling; in R. F. feebly yellowish while hot and colourless when cold. With S. Ph. behaves quite similarly.

Lanthanite from Bethlehem, Penn., becomes white when heated, then brown, and is infusible. With borax a bluish glass, growing brown on cooling and finally amethyst-red.

SILICATES.

a. Cerite yields water and becomes quite opaque in the matrass. On coal springs about and is infusible. Dissolves slowly in borax, giving in O. F. a deep, dark yellow glass, becoming lighter on cooling, and which can be flamed opaque. In R. F. the glass shows a feeble iron-colour. By S. Ph. the oxide of cerium is extracted with the usual play of colours; the glass appears colourless when cold, and a white opaque silica skeleton remains. Undissolved by soda, but half fused to a dark yellow slaggy mass. (Berzelius.)

b. Tritomite yields water and gives a feeble fluorine reaction in the matrass. B. B. burns white, swells somewhat, becomes cracked, and occasionally decrepitates with violence. Dissolves in borax in O. F. to a reddish-yellow glass, colourless on cooling. The finely pulverized mineral is decomposed by hydrochloric acid, with evolution of chlorine and separation of gelatinous silica.

c. Allanite yields in the matrass some water without changing its appearance; the water is not therefore chemically combined. B. B. fuses easily with intumescence to a black, lustrous, vitreous bead. Dissolves easily in borax to a black, opaque glass, which becomes blood-red, however, in O. F. while hot, and more or less dark yellow after cooling; in R. F. it assumes a fine, iron-green colour. Does not become opaque by flaming. S. Ph. decomposes it, leaving a silica skeleton. The glass shows the iron colour while hot, but in cooling becomes colourless and opalescent. (Berzelius.)

d. Orthite from Finbo and Gottliebsgang, as well as from granite near Stockholm and Söderköping, yields water in the matrass and becomes lighter in colour at a higher heat. On coal intumesces, becomes yellowish-brown, and finally fuses with ebullition to a black, blebby glass. Dissolves readily in borax to a glass, blood-red in O. F. and yellow when cold. In R. F. green. Is easily decomposed by S. Ph. with the usual phenomena. With a very little soda fuses; with more swells to a grayish-yellow slag. On platinum shows manganese. (Berzelius.)

e. Pyrorthite (containing one-third its weight of carbon) behaves, according to Berzelius, as follows :—

In the matrass yields very much water, the last portions being yellowish and having a burnt odour. The residue is as black as coal. Gently heated on coal and afterward ignited at one point, it takes fire and glows of itself. The combustion is more lively with several fragments, or with coarse powder, and when blown upon. It leaves the mineral white, or grayish-white, sometimes inclining to red, and so light and porous that it will not remain on the coal before the blowpipe flame. In the forceps it fuses with difficulty to a black bead with a dull surface. With borax and soda like orthite. Dissolves with difficulty in S. Ph., the porous mass remaining on the surface of the fluid bead, but sinking into it on cooling. It emerges again on being re-heated.

To detect the separate elements in the foregoing silicates a proper amount of the fine powder is treated with aqua regia in a porcelain vessel, evaporated to dryness, dissolved in water, and the silica filtered out. Ammonia is added in slight excess to the filtrate and throws down $\underline{\mathrm{F}}\mathrm{e}$, Ce, La, Di, and $\underline{\mathrm{A}}\mathrm{l}$; leaving most of the Ca, Mg, and Mn. By digesting the washed precipitate, after filtration, with a solution of potassa $\underline{\mathrm{A}}\mathrm{l}$ is separated, and can be obtained from the alkaline solution as directed for silicates under alumina. The metallic oxides freed from $\underline{\mathrm{A}}\mathrm{l}$ are treated while still moist with not too concentrated a solution of oxalic acid and warmed; this dissolves the iron, and the other oxides settle to the bottom in combination with oxalic acid, as a heavy crystalline powder. These salts are filtered out, washed with cold water, and ignited in the platinum capsule. The separation of the three oxides has been already described. After adding a little nitric acid to the filtrate, the oxide of iron can be thrown down with potassa and tested B. B. with borax. The further treatment of the lime, etc., has been given under the earths.

SILICATES CONTAINING TITANIC ACID.

Tscheffkinite behaves, according to G. Rose, as follows :—

In the matrass swells and yields a little water. B. B. glows at first, then intumesces strongly, becomes brown and finally fuses to a black bead. Its powder dissolves rather easily in borax to a clear glass, slightly coloured by iron; a small quantity yields a perfectly clear glass. In S. Ph. the same, but dissolves more slowly, and when much is added silica separates and the bead opalesces on cooling. Fuses with soda, but soon spreads out and sinks into the coal. By washing away the coal a few spangles of iron are obtained. On platinum shows manganese.

2. MANGANESE, Mn.

Its occurrence in the mineral kingdom and in metallurgical products.

Manganese forms an essential constituent of several minerals, differing considerably in chemical composition; it is found in the following combinations :—

a. With *arsenic;*
b. With *sulphur;*
c. As oxide either *free*, or combined with *water of hydration;*
d. With other *metallic oxides:*
    α. With *protoxide of cobalt;*
    β. With *oxides of zinc* and *iron;*
    γ. With *oxide of copper;*

e. Combined with *acids:*

- *α.* With *sulphuric acid;*
- *β.* With *phosphoric acid;*
- *γ.* With *carbonic acid;*
- *δ.* With *tungstic acid;*
- *ε.* With *tantalic* and *hyponiobic acids;*
- *ζ.* With *silicic acid* in silicates;
- *η.* With *arsenic acid;*
- *θ.* With *boracic acid.*

Manganese also forms a frequent constituent of various metallurgical products, occurring in the metallic state in raw iron and steel; with sulphur in the various matt-like products from the smelting of certain silver, lead, and copper ores, and especially also as protoxide with silicic acid in the different slags.

Examination for Manganese.

a. General examination for manganese.

Manganese can be very easily detected in substances containing, besides oxide of manganese, no other metallic oxides which give coloured glasses with borax and S. Ph. by simply dissolving them in those fluxes on platinum wire in the O. F. and then treating the bead with the R. F. The hot beads appear amethyst-red, but on cooling are red inclining to violet, and lose their colour when treated for some time in the R. F., especially on coal. S. Ph. is far less intensely coloured than borax, and the colour disappears much more readily in the R. F. Should a small proportion of other colouring oxides be present, they alter the amethyst colour obtained in O. F. slightly or not at all, but occasionally show their own peculiar colour after the manganese coloration has disappeared under the R. F., *e. g.*; sesquioxide of iron. Should the amount of sesquioxide of iron be large, the bead appears blood-red in the O. F. and yellow after short treatment in the R. F. When the amount of manganese is considerable, the assay should be quickly pinched together a little after the reduction, or else shaken off from the wire, so that it may cool

Oxides and Salts.

immediately and not give the protoxide of manganese an opportunity to colour the bead again by becoming more highly oxidized. If there is not manganese enough to colour the S. Ph. bead, the latter, after a sufficient quantity of the substance has been dissolved in it, is brought in contact with a little crystal of nitre, which causes it to froth up and assume on cooling an amethyst, or feeble rose-colour, according to the amount of manganese present. The small fragment of nitre is placed near by on a porcelain dish, and the bead, after being strongly heated on the wire in the O. F., is quickly brought against it, when the two salts unite and permanganate of potassa is formed by the resulting oxidation. The evolution of gas causes the glass to froth up, and it may show the red colour only when cold. On treating it again in the flame, the reaction produced by the nitre entirely disappears.

Another method must be employed to detect manganese in compounds containing more than a small proportion of distinctly colouring oxides. The best reagent is soda, which in all cases gives the most characteristic reaction for manganese. If there is not less than 0·1 per cent. of oxide of manganese present, the powdered substance is mingled with two or three volumes of soda and fused on platinum foil in the O. F. The oxide of manganese dissolves in the soda to a transparent, green mass, consisting of manganate of soda, which flows around the undissolved portion, and on cooling is distinctly bluish-green. If there is less than 0·1 per cent. of manganese, this green colour is not so easily obtained, but by employing two parts of soda and one of nitre all of the oxide of manganese is more highly oxidized, and the least trace of it colours the soda distinctly bluish-green, after the assay is perfectly cold.

To obtain a certain manganese reaction from garnets, Fischer has recommended to dissolve not too little of the mineral in a borax bead and then to fuse this with soda on platinum foil, when the manganese reaction is more distinct than with soda alone.

The presence of sesquioxide of chromium gives rise to yellow alkaline chromates when soda and nitre are used, but this in no way conceals the green colour of the alkaline manganate, since, when oxide of chromium is fused on platinum foil with equal parts of soda and nitre a very trifling quantity of manganese may be detected by the green colour of the perfectly cold, fused mass. This green is, however, no longer bluish, but yellowish-green.

Minerals containing any oxide of manganese, higher than the protoxide, evolve chlorine when heated with hydrochloric acid, and this can be detected by its odour.

Metallic compounds must be dissolved in nitric acid, evaporated to dryness, the salts decomposed by ignition, and the resulting oxides tested for manganese with soda and nitre, as above.

Metallic Arsenides and Sulphides.

If the substance consists of, or contains sulphides or arsenides of the metals, it must be roasted on coal before the above tests for manganese can be applied.

Dressed Ores.

When the substance contains silica and protoxide of cobalt at the same time, *e. g.*, an ore dressed in the large way, the above treatment with soda yields a blue mass, consisting of oxide of cobalt dissolved in silicate of soda, by which the green of the manganese is entirely concealed. After separating the silica and other injurious ingredients, however, by fusion with soda and borax and subsequent treatment in the wet way, as directed for silicates under lime, the manganese can be found with certainty.

b. Blowpipe characteristics of minerals of manganese.

ARSENIDE OF MANGANESE.

Kaneite burns on coal with a blue flame and yields a coat of arsenous acid (Kane). The residue undoubtedly gives manganese reactions.

SULPHIDE OF MANGANESE.

a. Alabandite is unchanged in the closed tube. In the open tube yields sulphurous acid and turns grayish-green on the surface. When thoroughly roasted on coal, which occurs very slowly, it reacts like pure oxide of manganese.

b. Hauerite yields sulphur in the closed tube and becomes green. In the open tube yields much sulphurous acid and becomes green on the surface. When well roasted, reacts like oxide of manganese with the fluxes.

OXIDES OF MANGANESE.

Most of the oxides, viz., *hausmannite, braunite, manganite, psilomelane, varvacite,* and *wad,* yield more or less water in the matrass, and such as contain a higher proportion of oxygen, especially *polianite, pyrolusite,* and *groroilite,* when heated to redness give off oxygen, which may be recognized by means of a little fragment of charcoal placed in the matrass.

They dissolve in borax and S. Ph., some of them with effervescence, produced by escaping oxygen, and either behave like pure oxide of manganese, or after reduction show iron. They frequently contain a small amount of alkalies, baryta, or lime, which may be detected by igniting them thoroughly in the O. F., laying them on platinum foil, moistening them with a few drops of water, and after some time testing this water with red litmus-paper. On dissolving such an oxide of manganese in hydrochloric acid, chlorine is evolved, and any silica present remains behind. The solution may then be further examined as directed under baryta and lime. *Pyrochroite* yields much water in the matrass, becomes green, then greenish-gray, and finally brownish-black.

OXIDES OF MANGANESE COMBINED WITH OTHER METALLIC OXIDES.

Black earthy cobalt from Saalfeld yields water, which has a burnt odour. B. B. in the forceps and on coal is infusible, but if $Co^3\dot{A}s$ is present, it gives a light blue flame, and on coal evolves a feeble odour of arsenic. With borax in O. F. a dark violet glass, smalt-blue in R. F. In S. Ph. only the cobalt colour, but the saturated bead treated on coal with tin becomes opaque-red on cooling (*vide* copper). Is not dissolved by soda, but gives a strong manganese reaction with soda and nitre.

The same mineral from Schneeberg yields water in the matrass. With borax and soda like the above; but with S.Ph. shows only the cobalt reaction.

Many so-called earthy cobalts contain so little Co and as much Mn that a very large amount must be dissolved in the borax bead and this treated for a long time in a good R. F. in order to produce a blue colour.

Franklinite alone is infusible; the moistened powder treated some time on coal in a strong R. F. yields a very distinct zinc coat. With borax and S. Ph. shows manganese; but the somewhat strongly saturated borax bead is rather red, and on coal in the R. F. becomes bottle-green from proto-sesquioxide of iron. With soda on platinum foil shows manganese, and on coal a slight zinc coat, becoming stronger when some borax is added.

Credneritc fuses only on the edges of very thin scales. With borax a dark violet glass; with S. Ph. a green glass, blue when cold, and becoming copper-red in R. F. Dissolves to a green solution in hydrochloric acid, with evolution of chlorine. (Rammelsberg.)

Lampadite, or *cupreous manganese*, yields much water in the matrass and then decrepitates somewhat. In R. F. on coal becomes brown, but is infusible. With the fluxes affords reactions for copper and manganese, and with soda and borax in R. F. on coal yields a little button of copper.

COMBINATIONS OF PROTOXIDE OF MANGANESE WITH ACIDS.

The phosphates of manganese, including *hureaulite, triplite, zwieselite, heterosite,* and *triphylite,* yield more or less water. B. B. fuse very easily to a globule and colour the flame. Those free from lithia give a bluish-green phosphoric acid flame; the others produce a red coloration at the same time. With the fluxes react for manganese and iron.

Megabasite behaves like *wolframite.*

Huebnerite.—In the forceps less fusible than *wolframite;* with the fluxes gives manganese and tungstic acid reactions. (Dana.)

Rhodochrosite and *manganocalcite* occasionally yield some water and often decrepitate very violently. Thoroughly ignited on coal and moistened with water, they generally have an alkaline reaction on red litmus-paper, owing to the presence of lime. They dissolve in the fluxes with effervescence, owing to escaping carbonic acid, and react like oxide of manganese containing iron.

Chondrarsenite decrepitates in the closed tube, blackens, and gives neutral water. On charcoal fuses easily to a black bead, not magnetic; in R. F. gives arsenical fumes. With borax a manganese reaction. (Dana.)

Sussexite behaves, according to Brush, as follows:—

In the closed tube darkens slightly and yields water, containing at least a trace of boracic acid. Fuses in the candle flame. B. B. in O. F. yields a black crystalline mass, and colours the flame intensely yellowish-green. With the fluxes affords the manganese reactions.

SILICATES.

Part of the silicates yield some water in the matrass, which occasionally has a burnt odour. Their fusibility is indicated by the annexed figures. With borax dissolve easily to a clear glass, showing manganese and more or less iron. With S. Ph. yield a silica skeleton and a manganese glass, generally colourless in the R. F., but sometimes opalescent on cooling.

They fuse with little soda to a black bead, with more yield a difficultly fusible slag, and an excess of soda goes into the coal. When it is necessary to determine any earthy admixtures, the method described for silicates under lime is followed.

c. *Examination for manganese in metallurgical products.*

In *raw iron* and *steel* manganese can only be found after solution in nitric acid, as directed for metallic compounds, p. 194.

Matt-like products, viz., *Rohstein*, *lead matt*, *copper matt*, etc., are powdered and thoroughly roasted on coal, after which the oxides formed are tested with soda and nitre according to p. 194.

The method of detecting manganese in dressed ores and slags has already been described under lime.

3. IRON, Fe.

Its occurrence in the mineral kingdom and in metallurgical products.

Iron is very widely spread throughout nature, occurring in most minerals, although sometimes only in traces. It is found under various conditions in the following combinations :

a. *Metallic*, or *meteoric iron ;*
b. Combined with *arsenic ;*
c. Combined with *arsenic* and *sulphur ;*
d. Combined with *sulphur ;*
e. With *phosphorus ;*
f. As *oxide*, eihter *free*, or *combined with water of hydration ;*
g. As *oxide combined with other oxides ;*
h. Combined with *acids*.

Iron is found under various conditions in the products obtained by smelting ores :

a. Metallic in

Raw iron and *steel*.

Bears (*Eisensauen*), which sometimes form in the shaft furnaces when smelting iron, copper, tin, and lead ores, owing either to some mistake in charging or to other causes, and which usually consist of a mixture of iron (carburet, siliciuret) and other metals, but very frequently contain an admixture of metallic sulphides and arsenides.

Black copper produced on the large scale, containing Cu as the chief ingredient, with more or less Pb, Ni, Co, As, Zn, Mo, Sb, Ag, is seldom free from Fe.

Finally, a little iron is found in unrefined tin, lead, and zinc.

b. Combined with *arsenic* in the various *speisses* produced in smelting lead, silver, and copper ores containing Fe, Ni, and Co combined with As.

The speiss produced in smelting roasted auriferous mispickel is a compound of Fe^4 As with Fe. The cobalt speiss from smalt-works consists chiefly of $(\text{Ni, Co})^3$ As, more rarely of $(\text{Ni, Co})^4$ As, with admixture of Bi; it sometimes also contains Fe^n As and Ág, more rarely Ꞓu.

c. Combined with *sulphur* in the various matt-like products from the smelting of gold, silver, lead, and copper ores.

in *lead matt ;*

in *copper matt*, consisting of $\overset{\prime}{\bar{\mathrm{C}}\mathrm{u}}$, $\overset{\prime}{\mathrm{Fe}}$, or $\overset{\prime}{\bar{\mathrm{C}}\mathrm{u}}$, $\overset{\prime}{\bar{\mathrm{F}}\mathrm{e}}$, and $\overset{\prime}{\mathrm{Fe}}$ in varying proportions, or combined with other metallic sulphides and arsenides.

Here belong also the *scaffolding, tutty* or *cadmia*, formed in the furnace by sublimation ;

Cadmia from lead furnaces, the chief constituent being Pb, which, however, often contains other sulphides, Fe, Zn, $\overset{\prime\prime\prime}{\mathrm{Sb}}$, and $\overset{\prime}{\mathrm{Ag}}$.

d. As *protoxide* with *silica* in the various slags.

e. As *proto-sesquioxide* in hammer-scales, forge-scales, etc.

f. As *protoxide* with sulphuric acid in copperas or green vitriol.

Examination for Iron,

Including the blowpipe characteristics of the minerals and metallurgical products above named.

a. Examination for iron in general.

This is very easy, since iron in combination with oxygen imparts a characteristic colour to borax and S. Ph., and cannot be separated from these fluxes in the metallic state by the blowpipe flame alone. It is only necessary to consider whether the substances treated are compounds of metals, or are metallic arsenides or sulphides, or finally metallic oxides.

Alloys. If they are alloys consisting only of difficultly fusible metals, they are fused beside borax on coal in O. F., until the glass is sufficiently coloured with the oxides of the easily oxidizable metals. Should lead, tin, bismuth, antimony, or zinc, be present, however, and the compound be easily fusible, the R. F. is employed, and is directed chiefly upon the glass, in order not to oxidize and dissolve too much of these metals. In both cases the still soft glass is removed from the metallic button and treated in R. F. on another spot, when the easily reducible metals are separated, leaving the borax coloured bottle-green by proto-sesquioxide of iron, provided oxide of cobalt does not prevent this reaction. If the compound contained tin, or the green glass is treated for a moment in R. F. with a bit of tin on a fresh spot of the coal, all the iron is reduced to protoxide and appears pure vitriol-green.

Should it, however, appear blue, protoxide of cobalt is present, which conceals the iron colour. In this case the glass must be again softened in the R. F., mostly removed from the coal without any adhering metal, and fused on platinum wire in a pure O. F. In case it should then become so dark as to be nearly opaque, the soft glass is pinched out, some of it broken off upon the anvil, and the remainder diluted with more borax. It is then again treated in the O. F. until all the iron is changed to sesquioxide, when it will colour the borax yellow to brownish-red, according to the amount present. Should there be, besides the cobalt, only a trace of iron, the hot glass will be green, but when cold pure blue.

A larger amount of iron colours the hot glass dark green and the cold glass fine green, since the sesquioxide, if not in excess, imparts a yellow colour to the cold borax glass, and this with the blue of the cobalt produces green.

The metals remaining after the treatment of the compound with borax in the R. F. may sometimes consist almost entirely of copper and nickel, since the volatile metals are mostly driven off and coat the coal with oxides, and they may be easily recognized by further treatment with borax or S. Ph., as will be directed under the examinations for the respective metals. If quite infusible compounds are to be treated, in which, besides iron and some of the above metals, nickel is also present, the safest way is to dissolve a little of the substance in nitric acid and continue the process as will be described under *native iron.*

Metallic Sulphides and Arsenides.

Compounds of metallic sulphides and arsenides may be examined for iron in two ways. In the first the assay is roasted on coal, and then small portions of it are gradually dissolved in borax on platinum wire in the O. F., and the colour of the glass examined, both when hot and cold. With many such compounds, containing only metals which do not colour very intensely when oxidized, the iron is immediately obtained; with many others, however, as when they contain copper, for instance, a green colour is obtained, which becomes lighter on cooling, and results from the yellow of the sesquioxide of iron and the blue of the oxide of copper. In this case the glass must be shaken off, and treated on coal in the R. F., until all the copper is reduced out and the bottle-green colour of the proto-sesquioxide of iron is obtained. After pinching out this glass, a bit of it may again be treated with the O. F. on platinum wire and the iron recognized by the yellow colour.

The second method consists in pulverizing the substance, mixing it with test-lead and borax and fusing it on coal in the R. F., until the glass is coloured by the easily oxidizable, non-volatile metals present. At first the whole is covered with the R. F., but as soon as the borax has united to one globule, the flame is directed upon this alone, allowing the air free access to the fusing metal. After completing the fusion the glass is quickly raised with the forceps from the fluid lead, and, after being treated alone on coal in the R. F. to reduce any trifling oxide of lead in it, is tested on platinum wire in the O. F. Should it appear too dark, it is diluted with borax until it is transparent. After separating the lead by means of boracic acid (*vide* copper), the other metals combined with it can easily be recognized by the glass fluxes.

Compounds which fuse easily alone on coal can be treated in the R. F. with borax, omitting the test-lead. Thus, for example, a very trifling amount of iron can be found in many galenas, especially if the glass is further treated with tin. Should the glass not show a vitriol-green colour, but be blue, it is treated as described above under alloys.

Compounds of Oxides, etc.

In compounds of oxides of iron with other metallic oxides, or with earths and acids, the iron is likewise best found by fusing the substance with borax or S. Ph. To determine whether the iron is present as sesquioxide or protoxide, the assay is added to a borax bead containing oxide of copper. In case of sesquioxide the bead becomes bluish-green; with protoxide, red spots of suboxide of copper become distinctly visible in it.

Compounds of metallic oxides not suspected of containing oxides of copper, nickel, chromium, or uranium, are dissolved in borax on platinum wire with the O. F., and the coloured bead held against the daylight and watched until it has so far cooled that its colour remains unchanged. The bead requires no further treatment if it shows only iron, or the colour of iron and cobalt together, as described before; should it, however, show some other colour, as possibly violet with much red, it must be treated some time in the R. F., which causes the violet colour, resulting from manganese, to disappear, and leaves the bottle-green iron colour. When much manganese is present, the bead from the O. F. appears quite dark-red while hot, and red, inclining to violet, on cooling, in which case all the manganese cannot be reduced to protoxide on the wire, but the glass must be shaken off and treated on coal with tin; the manganese colour then disappears and the vitriol-green of protoxide of iron becomes evident, provided no protoxide of cobalt is present. In case the manganese predominates, a little iron may also easily be found by means of S. Ph., which is not coloured very intensely by manganese, and readily becomes colourless in the R. F., while the colour of the dissolved oxide of iron remains after treating the glass in the R. F.: the glass usually appears reddish on cooling.

When a substance contains protoxide of cobalt, in addition to the oxides of iron and manganese, the glass obtained with borax on platinum wire in the O. F. is coloured more or less dark violet, and after a short treatment in the R. F. becomes green, and on cooling blue.

In a compound containing much oxide of manganese and cobalt with little oxide of iron, the latter may be readily found by dissolving the substance in hydrochloric acid and precipitating the sesquioxide of iron by means of ammonia from the diluted solution, or if the substance is not perfectly soluble, it is fused with bisulphate of potassa, the mass dissolved in water, a few drops of hydrochloric acid added, and then a slight excess of ammonia. The precipitated oxide of iron, although not quite free from manganese, is filtered out and tested with borax or S. Ph. on platinum wire.

When in addition to oxide of iron the oxides of copper and nickel are present, it is better to dissolve the substance on coal in borax with the O. F. and then treat it with the R. F.: copper and nickel are separated as metals and the iron colour alone remains. It is well to add a bit of lead, which furthers the separation of the metals; the glass can afterwards be pinched out and treated on platinum wire in the O. F., to obtain the pure colour of sesquioxide of iron.

Should the presence of cobalt render the glass blue, it must, in fact, be oxidized on the wire, as above directed. To discover the copper, the substance is dissolved in S. Ph. and the glass treated with tin on coal, when it becomes opaque and red.

When oxide of chromium is present with oxide of iron, the colour of the hot borax glass shows iron, but on cooling only chromium. Since, however, borax glass saturated with sesquioxide of chromium after treatment in the O. F. likewise has, while still hot, a dark-red colour, the presence of iron cannot with certainty be assumed. In such cases the substance is mixed with three parts of nitre and one of soda, and fused by degrees on platinum wire, the resulting chromate of the alkali dissolved in water, and the residue, after being washed with water, dissolved in borax on platinum wire. The iron colour is then obtained, if all the sesquioxide of chromium has been separated and no other colouring metallic oxides are present. The iron may also be reduced with soda on coal and obtained as metal after washing away the portions not reduced.

When uranium is present with iron, the borax, indeed, shows the iron colour; this, however, is not produced by the iron alone, but also by the similarly colouring uranium. To obtain the pure iron colour, the substance, if not completely soluble in acids, must be fused with bisulphate of potassa, the mass dissolved in water, and then an excess of carbonate of ammonia in solution added. The sesquioxide of uranium, which is at first thrown down with the sesquioxide of iron, dissolves again, so that the iron may be separated by filtration and tested with borax, after being washed. By boiling the ammoniacal filtrate the sesquioxide of uranium is thrown down as a yellow powder, and may likewise easily be recognized by testing it B. B. with S. Ph. An easier method of precipitating the uranium consists in slightly acidifying the solution with hydrochloric acid and then adding potassa.

Finally, when oxides of tungsten or titanium are present with the iron, only yellow iron beads are obtained with borax and S. Ph. in the O. F., because when combined with a maximum of oxygen the other metals (tungstic and titanic acids) only produce a feeble yellow; in the R. F., on the other hand, the S. Ph. glass assumes a very different colour, especially on cooling, it becomes darker or lighter brownish-red.

b. Blowpipe characteristics of the minerals containing iron.

Native iron and *meteoric iron* are infusible before the blowpipe. The glasses obtained with borax or S. Ph. on coal in the R. F. show only iron, and the bottle-green glass re-fused on platinum wire also shows iron alone. After dissolving the iron in nitric acid, diluting and then precipitating the sesquioxide of iron with excess of ammonia, nickel, cobalt, manganese, and copper can be thrown down by sulphide of ammonium from the ammoniacal solution, which contains the greater part of these metals present, and after settling they can be filtered out and recognized by means of borax. (*Vide* general examination for metallic sulphides, under cobalt.)

COMPOUNDS OF IRON WITH ARSENIC AND SULPHUR.

Leucopyrite (*arsenical iron*, pt.) yields in the closed tube metallic arsenic. Carefully heated in the open tube, much arsenous acid is sublimed, and with moistened litmus-paper sulphurous acid can be detected. On coal copious arsenical fumes are evolved and in the R. F. a magnetic globule remains. Roasted and treated with the fluxes it reacts only for iron. By treating the variety from Schladming on coal with borax, after being first fused alone, as will be directed under nickel for substances containing various metallic arsenides, some nickel and cobalt are found.

Mispickel (*arsenopyrite, arsenical pyrites*) yields at first in the closed tube a red sublimate of sulphide of arsenic, but later a black, crystalline sublimate of arsenic, having a metallic lustre: in the open tube gives off arsenous and sulphurous acids. Too strong a heat is apt to produce sublimates of suboxide of arsenic and metallic arsenic.

On coal, at first yields copious arsenic fumes and a coat of arsenous acid, then fuses, especially in the R. F., to a globule, which reacts like pyrrhotite.

In the roasted mineral any cobalt present is readily detected with borax. *Danaite* behaves like arsenopyrite, but reacts strongly for Co when roasted.

COMPOUNDS OF IRON WITH SULPHUR.

Pyrrhotite strongly heated in the closed tube yields a little sulphur; in the open tube only sulphurous acid. Fuses in R. F. on coal to a button, covered with an uneven black mass on cooling, magnetic and showing a yellowish, crystalline, metallic fracture. It is converted into red oxide by roasting and reacts with fluxes for iron only. Should it contain but little nickel, this is best found by treating the roasted assay with gold and borax in R. F. on coal (*vide* nickel).

Pyrite in the closed tube generally evolves an odour of sulphuretted hydrogen and gives a sulphur sublimate. If it contains arsenic, a sublimate of sulphide of arsenic forms later, which appears darker or lighter, according to the amount of arsenic. The well-ignited residue is metallic and porous, and reacts like pyrrhotite. On coal the sulphur burns with a blue flame and the residue reacts like pyrrhotite.

Marcasite behaves like pyrite, but yields sulphur at a lower heat, and moisture is frequently perceptible.

Lonchidite from the Churprinz mine, Freiberg, yields at first a sublimate of sulphur, in the closed tube, then a little sulphide of arsenic, which is reddish-yellow on cooling; in the open tube sulphurous and arsenous acids, at a high temperature sulphide of arsenic.

On coal in the R. F. sulphur and arsenic volatilize and an arsenic coat is formed; the assay then fuses quietly to a magnetic globule and a feeble lead coat is produced.

The roasted mineral dissolved in borax shows iron, cobalt, and copper with the fluxes. By a reduction assay with a gold button, which is afterwards treated with S. Ph., a little copper and cobalt can also be found.

Kyrosite from the Bricciusstolln, near Annaberg, reacts like lonchidite in the closed tube and the open tube.

On coal the sulphur burns and the mineral fuses to a magnetic globule without producing a noticeable coat of arsenous acid. The roasted powder is reddish-brown, and, treated with borax, shows iron and copper. The same metals are detected by means of S. Ph. By a reduction assay with soda on coal, metallic iron with an admixture of copper particles is produced.

OXIDES OF IRON AND HYDRATED SESQUIOXIDES.

The oxides, viz., *magnetite*, *ochreous magnetite*, *specular iron*, and *hematite*, behave in general like sesquioxide of iron. Trifling admixtures of other metallic oxides, viz., of chromium, maganese, copper, etc., may be either found at the same time by treating the respective oxides with glass fluxes, or by special assays, as directed in the corresponding passages.

The hydrates of sesquioxide of iron, viz., *turgite*, *limonite*, *gothite*, *xanthosiderite*, etc., with *clay-ironstone*, *bog ore*, and *ochre*, yield water in the matrass and change to sesquioxide, the red colour of which depends on the purity of the assay-piece. They fuse more or less easily on the edges in the forceps, particularly in the blue flame, while such as contain phosphoric acid tinge the outer flame bluish-green, this being observed with most certainty after moistening them with sulphuric acid. With borax and S. Ph. they all react for iron and sometimes for copper and cobalt. Clay-ironstone leaves a silica skeleton with S. Ph. A manganese reaction is obtained from nearly all of them, when fused with soda and nitre on platinum foil.

SESQUIOXIDE OF IRON COMBINED WITH OTHER OXIDES.

Magnesioferrite behaves like hematite (Dana).

Chromite.—B. B. in O. F. is infusible, in R. F. can sometimes be rounded on the edges, and is then magnetic. Dissolves slowly in borax and S. Ph. to a clear glass, showing iron while hot, but becoming chrome-green on cooling; the green is purer when the glass is treated in R. F. with tin on coal. It is not attacked by soda, nor can a manganese reaction be obtained on platinum; but if some nitre is added, the fused mass appears yellow from the formation of chromates of the alkalies. By a reduction assay, metallic iron is obtained.

OXIDES OF IRON COMBINED WITH ACIDS.

The sulphates of iron, viz., *melanterite* or *green vitriol*, *botryogen*, *glockerite*, *apatelite*, *copiapite*, *jarosite*, *coquimbite*, etc., yield more or less water in the matrass, which with a strong heat takes up some of the acid escaping from the assay and then has an acid reaction on litmus-paper. The salts containing protoxide of iron at first yield only sulphurous acid. On coal in O.F. they yield their acid and are converted into sesquioxide. The other characteristic ingredients, as copper in pisanite, zinc in roemerite, and the alkalies in *gelbeisenerz* and jarosite, may be found by the tests described under the respective substances. With soda they all give the sulphur reaction.

Halotrichite fuses in the matrass in its water of crystallization, swells up and yields much water. The residue heated to redness yields sulphurous acid and turns brown.

With the fluxes it shows iron, and with soda a sulphur reaction. If it is dissolved in water, and the protoxide converted into sesquioxide of iron by boiling with a little nitric acid, a precipitate of alumina and sesquioxide of iron is obtained by adding ammonia, and these may then be separated by means of potassa (*vide* alumina).

Pissophanite in the matrass yields water, which has, according to Erdmann, an acid reaction. The dry mass heated to redness yields acid vapours and becomes brownish-yellow on cooling. With the fluxes, shows iron; with soda on coal, gives an infusible hepatic mass. Cobalt solution only produces a distinct blue when the amount of iron is not too important. If the alumina cannot thus be found, it may be detected by dissolving the powdered mineral in hydrochloric acid and proceeding according to General Directions.

The phosphates of iron, *dufrenite*, *vivianite*, and *delvauxite*, yield in the matrass water, which has not an acid reaction. In the forceps they swell and fuse in the blue flame to a steel-gray metallic globule, producing a bluish-green phosphoric acid flame.

With the fluxes they show iron, and by a reduction assay with soda, or neutral oxalate of potassa, on coal, magnetic iron buttons are obtained.

Pseudotriplite behaves similarly, but shows a manganese reaction with soda.

In *alluaudite* the soda conceals the phosphoric acid flame, so that the latter must be shown in some other way (*vide* phosphoric acid).

As regards *calcioferrite*, it is only known that it yields water and fuses very easily to a black, lustrous, magnetic globule.

Diadochite yields much water in the matrass, increases in volume, and changes from brownish-red to 'yellow, losing its lustre and becoming opaque. Heated to redness it evolves sulphurous acid. In the forceps it expands very much and crumbles almost to powder. A fragment ignited in the matrass fuses in the forceps with intumescence to a globule, and colours the flame bluish-green (phosphoric acid). On coal it intumesces very strongly and then fuses to a globule, which glows while cooling. The cold globule is steel-gray and magnetic. With soda, nearly all goes into the coal; the fused mass yields a strong sulphur reaction, and by washing, magnetic metallic particles are obtained. With the fluxes, shows iron.

Cacoxenite yields water, the latter portions of which have an acid reaction on Brazilwood-paper; the liberated hydrofluoric acid attacks the glass, and rings of silica are seen after driving the water from the matrass. B. B., it fuses on the edges to a black, metallic slag, and gives a distinct phosphoric acid flame. Dissolves easily in borax and S. Ph., showing iron. With soda it at first fuses with effervescence, but afterwards the phosphate of soda sinks into the coal, leaving a black, infusible mass. It dissolves in hydrochloric acid, and leaves only a very slight amount of silica.

If the mineral is treated as directed for lazulite, the oxide of iron, alumina, lime, and silica can be found.

Childrenite yields much water. B. B., swells and ramifies, giving a distinctly bluish-green flame and forming a fissured, partly black and partly brownish-red mass, rounded on the edges. With the fluxes, shows iron and manganese (Rammelsberg).

The carbonate of iron, *siderite, spathic iron,* sometimes decrepitates in the matrass, gives off carbonic acid and oxide, blackens, and is converted into magnetic oxide. With the fluxes, like sesquioxide of iron. Soda sometimes produces a manganese reaction. Any lime or magnesia that may replace some of the iron can only be found in the wet way (*vide* carbonates, under magnesia).

Humboldtine yields water and blackens in the matrass. On coal it blackens, but in O. F. soon becomes red. With the fluxes, shows iron.

The arsenites of iron, including *arseniosiderite, pharmacosiderite,* and *scorodite,* yield neutral water in the matrass. B. B., in the blue flame fuse to a gray slag, with a metallic lustre, and colour the outer flame light blue.

On coal, give off arsenical fumes and fuse in R. F. to a gray, metallic, magnetic slag, which gives the iron reactions with the fluxes.

Pitticite yields water, and at a high temperature sulphurous acid. In the forceps and on coal, like scorodite. With soda, yields arsenical fumes and sinks mostly into the coal, giving a strong sulphur reaction on silver.

Carminite fuses on coal with evolution of arsenical fumes to a gray slag, and at the same time a lead coat is formed, or may certainly be obtained with the addition of soda. With the fluxes the iron reactions are produced.

Symplesite from Lobenstein heated to 100° C. is unchanged; above this heat it yields water (24·6 per cent.) and becomes brown; at a red heat a notable amount of arsenous acid is evolved, and a moistened slip of litmus-paper introduced into the neck of the matrass is feebly reddened.

B. B., it is infusible in O. F.; touched with the tip of the blue flame it fuses on the edges and colours the outer flame light blue. On coal in R. F. gives a strong arsenic odour and blackens, but fuses only on the edges and then is magnetic. After ignition in the matrass or on coal a fragment dissolves in the glass fluxes, showing iron, but in borax a good O. F. affords a somewhat brownish-yellow glass. If enough is dissolved on coal to make the borax glass quite opaque, subsequent reduction will produce fusible globules of a metallic arsenide, which may be collected by means of a gold button and tested in O. F. for nickel with S. Ph. (*vide* nickel).

With soda on coal, gives a strong arsenical odour, and the absorbed mass reacts feebly for sulphur. With soda and nitre, a slight manganese reaction.

Beudantite yields water. B. B., alone, the Cork crystals are infusible, but yield on charcoal fumes of sulphurous acid and afford a yellow slag, and with soda a kernel of lead; the Dernbach fuse easily on charcoal with intumescence to a globule of lead, mixed with a black hepatic slag; the Horhausen fuse easily, affording a gray slaggy globule, and after long blowing the odour of arsenic (Dana).

COMPOUNDS OF PROTOXIDES OF IRON AND MANGANESE WITH TUNGSTIC ACID.

Wolframite sometimes decrepitates in the matrass, and often yields traces of water. In the forceps and on coal, fuses with difficulty to a globule, the surface of which consists of accumulated lamellar, iron-gray crystals, having a metallic lustre. It is thus distinguished from titanic iron, which is infusible in the O. F.

Dissolves rather easily in borax in O. F. to a clear glass, in which the iron or manganese reaction predominates, according to the composition of the specimen, so that with a certain addition the varieties poor in iron can be distinguished from the richer ones; in the former the glass is rather reddish-yellow on cooling. In R. F. only the iron reaction.

With S. Ph. in O. F. shows only iron and manganese, but in R. F. becomes dark red and opaque, even with a moderate addition of the mineral. By treating the not too highly saturated glass on coal with tin in the R. F. for a very short time, it becomes green on cooling, but by then employing a good R. F. the green colour disappears, leaving a pale reddish-yellow, which remains unchanged. With soda and nitre it *gives a strong manganese reaction. For the detection of the* tungstic acid, *vide* tungsten.

COMPOUND OF PROTOXIDE OF IRON, ETC., WITH TITANIC ACID.

Menaccanite (*titanic iron*). Infusible in O. F., but can be somewhat rounded on the edges in R. F. With borax and S. Ph., in O. F. like sesquioxide of iron, but the S. Ph. bead treated a while in R. F. assumes on cooling a more or less intense brownish-red colour; by the depth of the red the relative amount of titanium can be estimated. On coal with tin this glass becomes violet-red, unless too little titanium is present. By fusion with bisulphate of potassa the mineral is decomposed (*vide* titanium). With soda and nitre it frequently gives a feeble manganese reaction.

Parathorite behaves, according to Dana, as follows :—

In the matrass decrepitates slightly. B. B. glows, fuses with difficulty on the edges, and becomes paler. In borax shows iron while hot, and is colourless on cooling. With S. Ph. in O. F. a bead, yellow while hot, colourless on cooling. In R. F. this bead assumes a delicate violet colour (TiO^2?); Brush.

COMPOUNDS OF PROTOXIDE OF IRON, ETC., WITH TANTALIC ACID.

Tantalite from Tammela, Kimito, and Finbo, free from tungstic acid, behaves as follows :—B. B. on coal and in the forceps it is infusible. Dissolves slowly in borax to a glass coloured by iron, which at a certain saturation becomes grayish-white by flaming, especially after previous treatment in R. F., owing to the tantalic acid. When fully saturated it becomes cloudy of itself on cooling. In S. Ph. dissolves also slowly to a bead coloured with iron, which treated in R. F. becomes pale yellow on cooling, but not red, showing that no tungstic acid is present. With coal on tin the glass becomes green.

With soda and nitre manganese is detected, and by a reduction assay with soda and a little borax, the latter serving to dissolve the tantalic acid compound and prevent the reduction of the iron, some tin can be obtained. If further evidence of the tantalic acid is desired, it may be obtained by proceeding as directed under tantalum.

Tantalite from Broddbo, containing tungstic acid, behaves like the above, except that the S. Ph. bead treated in R. F. becomes dark-red on cooling, and retains this colour when treated with tin on coal. Also gives a strong manganese reaction.

Tantalite from Kimito, having a cinnamon-brown powder, behaves, according to Berzelius, like a tantalite free from tungstic acid and containing manganese and some tin.

Tapiolite.—B. B. like tantalite, but no manganese reaction (Dana).

COMPOUND OF PROTOXIDE OF IRON, ETC., WITH HYPONIOBIC ACID.

Columbite is infusible. Dissolves easily in borax to a bead, coloured by iron, which can only be made opaque by flaming after strong saturation, and especially when first treated in R. F.

With S. Ph. like tantalite free from tungstic acid.

With soda and nitre it shows manganese, and by reduction on coal with borax and soda traces of tin are obtained, which with S. Ph. on coal frequently react for copper.

The manner of detecting hyponiobic acid will be given under niobium.

SILICATES.

Most of the silicates yield more or less water in the matrass. *Pyrosmalite* at a high temperature also gives off yellow sesquichloride of iron, which dissolves in the latter portions of water, and causes an acid reaction on litmus-paper; a suffocating odour is also perceptible at the mouth of the matrass.

Generally, if the blue flame has been used, the fused assay is magnetic. Some of them dissolve easily in borax, others with difficulty, and *anthosiderite* only very imperfectly, even in powder. The glass usually shows only iron; with S. Ph. they behave similarly, but those which have perfectly soluble bases leave a skeleton of silica.

With a little soda they fuse mostly to a bead, but with more, those that have a low ratio of silica fuse to a slag-like mass.

Several yield a manganese reaction with soda and nitre. To detect the earthy constituents in certain of these silicates, the method described under lime is to be followed.

c. *Examination for iron in metallurgical products, with the blowpipe characteristics of the latter.*

Raw iron and *steel* are usually only examined for accessory ingredients, viz., manganese, carbon, silica, sulphur, and phosphorus (*vide* the respective examinations).

The method of testing *bears*, *black copper*, and impure *lead* and *tin*, for iron and other accessory ingredients, is evident from what was said about metallic compounds containing iron.

Metallic arsenides.

The various *speisses* are very easily examined. They behave as follows:—In the open tube most of them yield sulphurous and arsenous acids, although certain of them must be first pulverized. On coal in R. F. they fuse to a globule and yield up their excess of arsenic, if the latter exceeds the proportion $(\mathrm{Ni, Co, Fe})^4\,\mathrm{As}$. Volatile metallic sulphides if present, as $\overset{\prime}{\mathrm{P}}\mathrm{b}$, $\overset{\prime\prime\prime}{\mathrm{S}}\mathrm{b}$, form a coat of oxides of lead and antimony, the latter being mixed with sulphate of lead. Should there be so much iron that the coat forms with difficulty, the iron must first be mostly removed by treatment with borax on coal, and the remaining button will then yield a distinct coat when treated alone. If the speiss contains bismuth, as is the case with cobalt speiss, a bismuth coat is obtained.

When the fused button of speiss is treated with borax on coal, iron oxidizes first, then cobalt and the resulting oxides dissolve at once, while arsenic volatilizes and is perceived by the odour. As soon as the button shows a bright surface the blast is stopped, the button quickly lifted out, and a portion of the soft glass withdrawn with the forceps, and if quite opaque, treated on platinum wire in the O. F. with borax. It will show either iron alone, or cobalt likewise.

The button is to be treated on coal with fresh borax, when, if all the iron and cobalt were removed before, the glass will show only nickel; but if some cobalt still remained, the glass will be coloured by it also; and if there is much cobalt, will be, indeed, pure smalt-blue. In this case still a third, or even a fourth treatment with borax may be necessary, and then only the nickel colour will be observed.

Should the speiss contain copper, this metal would not be detected by means of borax, being less easily oxidized than nickel, but may be very readily found if the button, freed from iron and cobalt and now containing only $Ni^4 As$, with with more or less $\acute{C}u$, is treated on coal in the O. F. with S. Ph. Copper and nickel are then oxidized, and the glass bead is yellowish-green, retaining this colour on cooling, owing to the yellow of the nickel and the blue of the copper. Treated with tin on coal, this bead becomes red and opaque from suboxide of copper, when cold. It is assumed that all the antimony has been previously removed by treating the speiss alone on coal, so that the bead shall not become black on cooling.

When there is so much sulphide of lead in the speiss that the coat of oxide of antimony cannot well be distinguished from the simultaneously formed sulphate of lead, it is only necessary to treat the powdered speiss with soda in the R. F. The sulphur is separated by the soda, and the lead then forms only a yellow coat, allowing the oxide of antimony to form a pure coat. When there is a considerable amount of sulphide of zinc, a slight zinc coat is also formed; but if the amount of zinc is trifling, it cannot always be shown with certainty.

In treating a very impure speiss, containing many sulphides, a little of it may, after the presence of volatile metal has been ascertained by treatment on coal, be well roasted, and then treated with the fluxes as described under general examination for iron.

Metallic sulphides.

The various matt-like products evolve in the open tube sulphurous acid and, if they contain sulphide of antimony, deposit near the assay a thin, fixed sublimate of oxide of antimony and antimonic acid. On coal in R. F. they fuse to a globule, with the exception of *Rohofenbruch* rich in zinc, and coat the coal with oxides of lead, antimony, and zinc, and sulphate of lead, when they contain volatile sulphides of these metals, and have not very little sulphide of zinc. Occasionally also the odour of arsenic is perceptible; otherwise a special test may be made for it (*vide* arsenic).

To detect the other ingredients, a sufficient amount is roasted on coal and tested first with borax and S. Ph., as directed for the compounds of oxides of iron with other metallic oxides. Another roasted portion is treated in the R. F. with soda, so as to produce metallic iron and copper, and to recognize any small amount of zinc by the coat which is formed in the immediate neighbourhood of the assay.

Slags vary so much that it is not possible to establish any general blowpipe characteristics for them, but it is very easy to find, by means of their behaviour alone on coal and with the glass fluxes, what metallic bases they contain, and regard must be had to these when effecting their decomposition, partly in the dry way and partly in the wet way.

Hammer and *forge scales,* from working wrought iron, are immediately recognized by the fact that they fuse to a bead in the forceps, when touched with the blue flame, and react only for iron with the fluxes. Sometimes a manganese reaction can be obtained with soda and nitre.

4. COBALT, Co.

Its occurrence in the mineral kingdom and in metallurgical products.

Cobalt occurs under different conditions in the following minerals:

a. Combined with *arsenic;*
b. Combined with *arsenic* and *sulphur;*
c. Combined with *sulphur;*
d. Combined with *selenium;*
e. As *oxide* and combined with other *metallic oxides;*
f. Combined with *acids.*

In metallurgical products from ore containing cobalt as an essential, or only as an accidental ingredient, this metal is likewise present. In addition to *smalt* from the smalt-works, the following are the chief products to be named:

Cobalt speiss, which settles in the pots during the preparation of smalt.

Nickel speiss, obtained in smelting nickel ores poor in cobalt, to concentrate the arsenides of nickel and cobalt.

Lead speiss, resulting when ores containing silver, cobalt, nickel, lead, and copper, are smelted with plumbiferous materials, and consisting chiefly of $(Fe, Ni, Co)^4 As$ with the bases in very variable proportions.

Refined speiss, so far freed from accessory ingredients by concentration and refining, that it approaches the composition $(Ni, Co)^4 As$.

Rohstein, lead matt, copper matt (*vide* iron).

Slags from smelting ores and products containing cobalt, and also from refining niccoliferous and cobaltiferous black copper.

Examination for Cobalt.

a. General examination for cobalt.

Cobalt is very easily detected, since it oxidizes quite readily and then imparts a smalt-blue colour to the borax and S. Ph. beads, which remains the same in both O. F. and

R. F. Simple and compound substances cannot, however, be treated in similar ways.

Metallic nickel.

Metallic nickel, which is infusible, is converted into arsenide before testing it for cobalt, by mixing it in thin scales, or filings, with a little metallic arsenic, fusing them together in a cavity on coal with the R. F. and treating the fused button a short time with borax directly with the tip of the blue flame; if any cobalt is present, the glass becomes blue, and if the amount is not too trifling, the cleansed button will impart a blue colour to a fresh portion of borax also.

Compounds of metals

The manner of finding cobalt in alloys has been described under iron.

Metallic arsenides.

Compounds of cobalt with arsenic and other metallic arsenides are fused on coal until they cease to evolve arsenic, and then borax is added and fused with the metallic compound, now containing less arsenic, until the glass is coloured. It will be pure smalt-blue, unless iron is present, which oxidizes sooner than cobalt, and produces, at the same time, the colour of its proto-sesquioxide. The cleansed button treated with fresh borax will, however, show the pure cobalt blue. Any nickel and copper present will be combined with arsenic or sulphur, and do not oxidize until all the cobalt has been separated by repeated fusions with borax in O. F. When the fresh borax no longer assumes a blue colour, but is brown from nickel, the remaining button is treated with S. Ph. in O. F. and the glass becomes green, both when hot and cold, if copper as well as nickel is present. On coal with tin it becomes opaque and red from suboxide of copper.

Any bismuth is immediately recognized by the coat formed while removing the excess of arsenic by treating the substance alone on coal. If no antimony is present the coat may be tested with S. Ph. and tin (*vide* bismuth). Metallic arsenides in which cobalt forms a chief constituent may be roasted and tested with the fluxes as directed for sulphides, but the above method is always the shortest.

Metallic sulphides.

Sulphides, sometimes containing arsenides, are first treated alone on coal in R. F. until they cease to yield anything volatile. The coats then formed will indicate any admixture of lead or bismuth. The fused compound is powdered, well roasted, and a part of it at once tested on coal with borax in O. F. If no colouring oxides except cobalt are present, the glass will be blue, and remain so when diluted with borax and tested on platinum wire in O. F. A trifling amount of iron will, however, then render the glass green while hot. If copper or nickel is present their oxides will likewise dissolve and sometimes entirely conceal the cobalt colour. By treating such a glass on coal, however, in the R. F., until it appears transparent while fused and few or no bubbles escape from it, the copper and nickel are reduced to metal, and either the pure cobalt will appear, or the cobalt colour mingled with the bottle-green of iron. The separation of the metals is promoted by adding a little test-lead, but then the metallic compound obtained should be freed from the excess of lead by treating it alone on coal, after which it is fused in O. F. with S. Ph. to detect nickel and copper. Gold may be used in place of the lead (*vide* nickel).

Metallic selenides.

Metallic selenides are first treated alone and then with borax on coal in R. F., until the glass is coloured by the easily oxidized, fixed metals. Should the glass not show a pure cobalt colour, it is treated as above described.

Oxides and salts

In treating metallic oxides or their salts, in which protoxide of cobalt forms a chief, or accessory ingredient, a small quantity is fused with borax on coal in R. F., until all the non-reducible oxides are dissolved, while the others are reduced to metal, and such as are volatile have been volatilized. If oxides of cobalt, iron, and manganese are present, the iron dissolves as proto-sesquioxide, and the manganese as colourless protoxide, so that the glass has a mixed blue and bottle-green colour, very easily distinguished from the green produced by iron alone in R. F., even if little cobalt is present. This glass in O. F. on platinum wire only shows cobalt and iron distinctly when manganese is absent; otherwise the manganese becomes more highly oxidized and, colouring the glass intensely, conceals the cobalt.

The metals separated by treating the glass in R. F. may form a button, *e. g.*, when much arsenate of nickel is present, and they can be further tested with borax and S. Ph. Should the amount of nickel be so small as to give no certain result in this way, another method must be employed, as will be specially described under nickel.

b. Blowpipe characteristics of minerals containing cobalt.

COMPOUNDS OF COBALT WITH ARSENIC.

Smaltite usually yields in the closed tube metallic arsenic. Carefully heated in the open tube, an abundant crystalline sublimate of arsenous acid is obtained, and sometimes sulphurous acid. In powder it is converted into basic arsenate of cobalt. It fuses on coal, with evolution of arsenic fumes to a grayish-black, magnetic, metallic button, which is brittle, and with borax behaves like arsenide of cobalt containing a little iron and nickel.

Skutterudite gives a strong sublimate of arsenic in the closed tube, otherwise like smaltite.

Wismuthkobaltzerz in the closed tube yields metallic arsenic; in the open tube a sublimate of arsenous acid, and with litmus-paper shows sulphurous acid. On coal *sinters*, yields much arsenic, and forms a bismuth coat. By treating the remaining mass with borax, and the button which then separates, after freeing it from cobalt, with S. Ph., iron, cobalt, copper, and nickel can be detected.

COMPOUNDS OF COBALT WITH ARSENIC AND SULPHUR.

Cobaltite (*glance cobalt*) yields in the closed tube only a very little arsenous acid formed by the air in the tube. In the open tube at a red heat it yields arsenous acid and sulphurous acid. On coal it yields sulphur and arsenic, and fuses to a button, which reacts with borax like arsenide of cobalt containing iron. By treating it further with borax, adding gold to increase its volume, any nickel present may be detected.

Glaucodot loses its lustre in the closed tube; otherwise like cobaltite in the closed and open tubes.

It fuses quietly on coal in the R. F., yielding sulphur and arsenic to a button, which on cooling has a black, rough surface, but a fine-grained, speiss-like fracture, and is slightly magnetic.

This button, treated with borax, first shows a strong iron reaction; with fresh borax only pure smalt-blue, and if finally gold is added to increase the volume of the little button remaining, and the treatment with borax continued, the last traces of the metallic arsenides oxidize and show a feeble nickel reaction.

COMPOUNDS OF COBALT WITH SULPHUR.

Syepoorite.—The blowpipe characteristics of the natural mineral are not known. The artificial yields sulphurous acid in the open tube; nothing in the closed. On coal it fuses to a globule, which even after long treatment with the R. F. still shows a bright surface on cooling, and is magnetic. The roasted powder shows pure cobalt reactions with the fluxes.

Linnœite from Siegen yields a slight sulphur sublimate in the closed tube; and much sulphurous acid in the open tube, with a very little arsenous acid. If used in powder, it becomes black on cooling. On coal, small fragments of crystal fuse in the R. F., with some evolution of sulphur, to a globule, which can be kept fluid for some time with the surface free from oxide, and forms no coat. When cold it is covered with a black, rough oxide film, probably magnetic oxide of iron, and both the whole globule and fragments free from oxide follow the magnet.

The roasted powder affords reactions for cobalt, iron, and nickel. By reducing some of the roasted powder with neutral oxalate of potassa, a magnetic metallic powder is obtained. (Had the metals been combined partly or entirely with arsenic, the roasting would have formed basic arsenates, and by reduction globules of metallic arsenides would be produced.)

COMPOUND OF COBALT WITH SELENIUM.

Tilkerodite yields in the closed tube a sublimate of selenium. On coal, evolves the odour of selenium, coats the coal with selenium and oxide of lead and tinges the flame azure-blue.

The assay decreases in volume, without fusing perfectly, and leaves finally an unalterable scoria, which gives iron and cobalt reactions with borax.

COMPOUNDS OF PROTOXIDE OF COBALT WITH ACIDS.

Bieberite yields in the matrass water and, on continued heating, sulphurous acid. With the fluxes it reacts like oxide of cobalt. The magnesia can only be found by dissolving the salt in water and then separating the bases.

Erythrite yields in the matrass only water. The red crystals from Schneeberg glow, and on cooling are dark, dirty violet. B. B. in the forceps the crystals fuse and colour the flame light blue. On coal it evolves arsenical fumes and fuses in R. F. to a blackish-gray globule of arsenide of cobalt, which with the fluxes reacts only for cobalt.

Earthy cobalt bloom yields in the matrass water and arsenous acid. On coal and with fluxes, like erythrite.

Lavendulan yields only water. The assay becomes lamellar, and is bluish-gray when cold. In the forceps fuses easily and colours the flame light blue. The fused assay crystallizes with large faces on cooling, like phosphate of lead. The crystals are generally black and opaque, but some have a dark hyacinth-red colour. On coal in R. F. it fuses, and seems to be reduced, while a strong arsenical odour is perceptible. With the fluxes, shows cobalt, nickel, and copper.

c. Examination for cobalt in metallurgical products.

The method of proceeding may be deduced from the examination for iron in general, and for products of iron.

5. NICKEL, NI.

Its occurrence in the mineral kingdom and in metallurgical products.

Nickel occurs under different conditions:

a. Combined with other *metals;*

b. Combined with *arsenic;*

c. With *antimony*, *arsenic*, and *sulphur*;
d. Combined with *sulphur*;
e. In the *oxidized state*;
f. Combined with *acids*.

Small quantities of nickel are also found in
Chrysolite and olivine (*vide* magnesia);
Meteorites (*vide* iron);
Chrysoprase (*vide* quartz and silicic acid).

Several metallurgical products contain nickel, when obtained from niccoliferous ores. It is usually concentrated in combination with arsenic and arsenides of cobalt and iron, either in the sulphide compounds, (matts, regulus) obtained from smelting certain silver, lead, and copper ores, and then forms only an admixture (*vide* iron); or it settles in combination with arsenic and other arsenides, as well as with metallic sulphides, as a special product, viz., speiss, lead speiss (*vide* cobalt). It further forms the chief ingredient of the speiss obtained by smelting cobaltiferous nickel ores, to concentrate the arsenides of cobalt and nickel in the ores, as well as of refined speiss and cobalt speiss. Occasionally also it forms an accessory ingredient of black copper, obtained in the large way, and of certain slags.

Examination for Nickel.

a. General examination for nickel.

Nickel can be detected with certainty and comparative ease, even when in very trifling quantities. For its reactions with the fluxes, *vide* tables.

Alloys.

Fusible alloys containing nickel are melted for some time with borax on coal in R. F. and the glass tested on platinum wire in O. F., notice being also taken of any coat formed on the coal. The remaining metallic button is again tested with borax in R. F., to ascertain whether the glass still takes up any oxides of the non-reducible metals, or whether it remains colourless. In the latter case the button is treated with S. Ph. in O. F., to discover whether only the nickel coloration results, or whether copper is likewise present, in which case a green, or yellowish-green glass is formed, which remains green on cooling, and with tin on coal becomes opaque and red. Should antimony or bismuth, however, be also present, the glass bead becomes black on cooling, and the copper reaction is thus concealed. It is then necessary to employ a new piece, and before using the fluxes, to treat it alone on coal in R. F., until it ceases to yield anything volatile.

When the alloy is infusible, and consists especially of iron, the process indicated for native iron may be followed; but when it seems to consist chiefly of nickel and cobalt, these may be converted into arsenides for testing metallic nickel or cobalt.

Arsenides and sulphides

Compounds of nickel with arsenic and arsenides, or sulphides, which sometimes contain arsenides, are treated just like the corresponding cobalt compounds.

Metallic oxides and salts.

In metallic oxides and their salts, the nickel, if not in too small quantity, can be found by the method given for the corresponding cobalt compounds, but a very trifling amount cannot always be thus detected with certainty. It is then safer to proceed as follows:—Suppose it is desired to test a combination of oxides of cobalt, manganese, and iron for a trifling amount of oxide of nickel; a sufficient quantity should be dissolved in borax on platinum wire in O. F., the very dark, or quite opaque bead shaken off, and two or three such beads prepared. These are treated in a cavity on coal, or in a coal crucible, with a pure gold button of fifty to eighty milligr. weight, in a strong, active R. F., until it is certain that all of the nickel is reduced from the bead and collected in the gold button, which has been brought into contact with every portion of the fluid glass by carefully turning the coal. When the button has solidified, it is lifted from the glass and freed from any adherent glass between paper on the anvil. A trifling amount of nickel suffices to render the button more or less gray and harder under the hammer than pure gold. If the borax-glass was not supersaturated with oxides, so that none of the cobalt could be reduced, the gold button treated for some time in O. F. on coal with S. Ph. will impart to this only the nickel colour; reddish to brownish-red while hot, and yellow to reddish-yellow after cooling, according to the amount dissolved. If, however, cobalt had been reduced, it will oxidize sooner than the nickel, and either produce a blue cobalt bead only, or a bead which will be dark-violet when hot and dirty green on cooling, if some nickel had been oxidized. In either case the button, freed from glass, is treated with fresh S. Ph. in O. F., until the hot glass seems coloured, when, if the original borax beads had not been too highly supersaturated, the glass will show only the nickel coloration; if the metallic oxides were, however, free from nickel, the glass will be colourless.

When the oxides or salts contain other colouring oxides which are likewise reduced in the metallic state from the borax beads, as oxides of copper, the gold button will contain both nickel and copper, and, after being freed from any trifling amount of cobalt by means of S. Ph., will yield with a fresh portion of that salt a glass which is green while hot, even if the copper predominates considerably, and remains green on cooling, but treated with tin becomes red and opaque. If the assay was perfectly free from nickel the hot S. Ph. bead will, indeed, be green, but it becomes blue on cooling.*

b. Behaviour of niccoliferous minerals before the blowpipe.

COMPOUNDS OF NICKEL WITH ANTIMONY, ARSENIC, AND SULPHUR.

Melonite (*vide* under tellurium).

Breithauptite in the open tube evolves copious antimonial fumes, without fusing; the assay is grayish-green on cooling. It fuses on coal in R. F., and after the blast is stopped continues to emit fumes for a short time, like antimony, but without becoming covered with oxide of antimony. On renewing the blast a coat of oxide of antimony is formed, near which a yellow lead coat may be produced by disseminated galena. Should the mineral alone give no arsenic odour, this becomes perceptible on adding soda, and the soda will also afford a sulphur reaction in case the assay was not quite free from galena. The glass obtained with borax on coal in R. F. shows only iron, and also when this glass is afterward treated on platinum wire in O. F. only an iron coloration is produced; but by treating the remaining button with fresh borax in O. F., the nickel reaction is obtained.

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* A gold button containing nickel or copper is purified by fusing it with a suitable amount of test lead, cupelling it on bone-ash, and, if necessary, treating it with boracic acid on coal.

Niccolite, free from antimony, yields a very little arsenous acid in the closed tube, but in the open tube it yields arsenous acid abundantly and sometimes sulphurous acid; the assay becomes yellowish-green and crumbles to powder. On coal it yields arsenical fumes and fuses to a button, which treated a short time with borax shows cobalt and iron, and these are very distinctly seen when the glass is remelted on platinum wire in O. F. A feeble lead or bismuth coat is sometimes formed, and the remaining button shows nickel reactions.

Tombazite behaves like niccolite containing a little Co and Fe.

Rammelsbergite and *chloanthite* behave like niccolite, but yield metallic arsenic in the closed tube; the residue then corresponds to niccolite. With the fluxes a little cobalt and iron can generally also be detected, while a trifling bismuth coat is occasionally formed.

Ullmannite (*niccoliferous gray antimony*) yields a trifling white sublimate in the closed tube, and in the open tube copious antimonial fumes and sulphurous acid. On coal in R. F. fuses to a globule and evolves antimonial fumes, which partly coat the coal. Sometimes arsenic replaces part of the antimony, and may be detected by its odour, which is most perceptible when the assay is fused in O. F. with test lead (*vide* arsenic).

With the fluxes, iron, cobalt, and nickel are detected as described under niccolite.

Gersdorffite (*nickel glance*) decrepitates in the closed tube and yields a yellowish-brown sublimate of sulphide of arsenic. In the open tube arsenous and sulphurous acids. On coal yields sulphurous and arsenical fumes and fuses to a globule, which gives with the fluxes iron, cobalt, and nickel reactions (*vide* niccolite).

Amoibite reacts probably like the above, while ullmannite, when containing arsenic as well as antimony, as in *Antimon-Arsennickelglanz*, gives the reactions of both ullmannite and gersdorffite.

Millerite yields sulphurous acid in the open tube. On coal fuses rather easily to a globule, which spirts strongly and diminishes somewhat in volume, but remains fluid. Roasted and then treated with a good R. F., it yields a coherent, somewhat malleable, metallic, and magnetic mass. Well-roasted millerite gives the nickel reactions with the fluxes, but frequently a little iron and copper can be detected.

Grunauite in the open tube yields sulphurous acid and a slight yellowish-white coat, apparently of sulphate of bismuth, which settles near the assay. On coal it evolves sulphurous acid and fuses to a gray button, which afterward affords a yellow coat of oxide of bismuth and a white coat of the sulphate. The residue powdered, roasted, and treated with the fluxes, shows nickel chiefly, with considerable quantities of cobalt, copper, and iron. Lead cannot be found in the dry way alone, on account of the not unimportant amount of bismuth.

Pentlandite in the open tube yields sulphurous acid. On coal fuses to a globule, having a yellowish, metallic fracture. Roasted and tested with borax, shows chiefly nickel and iron, with a little copper from disseminated chalcopyrite.

PROTOXIDE OF NICKEL.

For its reactions *vide* tables.

PROTOXIDE OF NICKEL COMBINED WITH ACIDS.

Zaratite at 100° C. yields much water in the matrass and assumes a black colour. It dissolves with effervescnce in the fluxes and behaves like protoxide of nickel.

Annabergite in the matrass yields water and becomes darker. B. B. fuses in the tip of the blue flame and tinges the outer flame light blue (arsenic). On coal in R. F. yields arsenical fumes and fuses to a blackish-gray globule of arsenide, which usually with borax in R. F. first shows cobalt feebly, and then in O. F. with fresh borax shows nickel.

With soda on coal occasionally yields the sulphur reaction.

Arsenate of nickel.—Both varieties react like annabergite, but are unaltered in the closed tube (Dana).

B. B. fuses only on thin edges, and then assumes a gray appearance. Dissolves rather easily in borax and S. Ph., showing nickel and silica. It is imperfectly dissolved by soda; if the R. F. is employed, magnetic metallic nickel is obtained upon washing away the slag. The remaining silicates behave quite similarly.

To detect the magnesia, lime, alumina, and trifling amount of iron in these minerals, they must be powdered and fused with soda, borax, and silver on coal in R. F., as directed for silicates of lime.

Morenosite yields acid water in the matrass, swells up and hardens, becoming yellow and opaque. With borax and S. Ph. a distinct nickel reaction. The Riechelsdorf mineral colours the outer flame blue, from the presence of arsenic (Dana). A sulphur reaction is obtained with soda in R. F.

c. *Examination for nickel in metallurgical products.*

The method of examining products for nickel has been given partly under the general examination for iron, and partly under the description of products containing iron.

6. ZINC, Zn.

Its occurrence in the mineral kingdom and in metallurgical products.

Zinc occurs in nature under the following conditions:

a. *Metallic;*
b. Combined with *sulphur;*
c. In a combination of *sulphide* with *oxide;*
d. As *oxide;*
e. As *oxide* combined with other *metallic oxides;*
f. Combined with *sulphuric acid;*
g. Combined with *carbonic acid;*
h. Combined with *arsenic acid;*
i. With *silicic acid;*

k. Combined with *alumina.*

Zinc occurs in various metallurgical products:

a. Metallic in *Raw zinc.*

The first portions distilled from cadmiferous ores are particularly rich in cadmium.

b. Combined with *sulphur* in various products consisting of metallic sulphides, viz., lead and copper matts, cadmia from lead-furnaces, when these products result from silver, lead, or copper ores containing blende.

c. As *oxide,* which collects at the commencement of the zinc distillation in the condensers, together with metallic zinc, as well as during the further progress of the operation; the first portions are usually very rich in cadmium; the oxide also is found in the slags and flue rakings produced by smelting roasted silver ores containing blende in shaft and reverberatory furnaces.

Here is included also the cadmia of the iron blast-furnaces, which sometimes consists of pure crystallized oxide of zinc, but frequently forms only a compact mass of oxides of zinc and iron mixed with earthy particles.

Examination for Zinc.

a. General examination for zinc.

The examination for zinc is very simple, as the metal is volatile, while its oxide is fixed in the O. F., and is very certain in case the substance contains much zinc, or if containing little zinc, is free from other metals or oxides, which are reduced on coal and form a coat. When, however, a very small amount of zinc is present with much lead, antimony, or bismuth, for example, it cannot always be certainly detected by the blowpipe.

Metallic Sulphides, Oxides, etc

Substances containing much zinc, either as sulphide or oxide, are treated alone, but those containing only a little may be powdered and fused with sufficient soda on coal in R. F.; generally, however, the latter will also afford a distinct zinc coat with a good R. F. alone. When the substance is a combination of metallic oxides, or contains possibly some earths in addition, a mixture of two parts of soda with one to one and a half of borax is employed. The zinc is thus volatilized as metal, but immediately oxidizes again, and deposits on the coal a coat, which is yellow while hot and white when perfectly cold. This coat is especially characterized by the green colour which it assumes when moistened with cobalt solution, and ignited in O. F. If the substance contains much lead, the zinc coat, although not so far from the assay as the lead coat, is usually rendered impure by oxide of lead. On moistening such a coat with cobalt solution and carefully igniting it in O. F., the oxide of lead is reduced by the glowing coal and volatilizes, leaving the oxide of zinc, which assumes a green colour on cooling. Should the zinc coat be so thin that it is liable to be blown away after being moistened with cobalt solution, a decisive result may not always be obtained, and it is then better to moisten the coal where the zinc coat generally forms with the solution before treating the substance with the flame. A single drop spread out with a glass rod is sufficient for detecting a little zinc. Since it is necessary to treat the substance for some time with the blowpipe flame, the moistened spot is ignited, at the same time, any oxide of lead or bismuth mixed with the oxide of zinc is removed, and the deposited zinc coat appears distinctly green when cold. A similar process may be used in case of a feeble coat already formed by moistening the latter, and then directing the flame not upon it, but directing the R. F. again upon the assay on the coal, whereby more zinc is volatilized, while the already moistened coat is ignited throughout.

It must be borne in mind, however, that when the substance contains little or no zinc, but much antimony, and the coal is moistened before employing the blowpipe, a combination of cobalt with one of the acids of antimony will be formed, which likewise has a green colour and cannot be driven off with the O. F. In this case a little zinc can only be found with difficulty before the blowpipe.

With many compounds, however, as in antimonial tetrahedrite, it is possible first to volatilize nearly all of the antimony with the O. F. and to remove the oxide of antimony from the coal by directing the flame upon it, after which the zinc may be detected by treating the residue in the R. F. as above. When tin is present, zinc cannot be recognized by the coat formed on coal, as its oxide is then mingled with binoxide of tin, which assumes a bluish-green colour with cobalt solution.

b. Behaviour of zinciferous minerals before the blowpipe.

COMPOUNDS OF ZINC WITH SULPHUR.

Sphalerite in the closed tube sometimes decrepitates with great violence, but yields nothing volatile and generally retains its colour. The ignited assay being strongly heated in the open tube evolves sulphurous acid, and if heated for a sufficient time appears yellowish or brownish-red, according as it contains little or much iron.

Treated alone in R. F. on coal, it first yields a feeble, reddish-brown coat of oxide of cadmium, unless too little cadmium is present, but afterward a distinct zinc coat: it is infusible. In O. F. it roasts completely, but rather slowly, and then with borax readily shows whether much or little iron is present. With soda on coal it affords zinc and cadmium coats.

Leberblende yields in the matrass water and a trifling sulphur sublimate; at first an odour of sulphuretted hydrogen is also evolved, which afterward changes to a burnt odour. It decrepitates violently and becomes more or less black. The variety from Cornwall becomes nearly black; but there are varieties which, on the contrary, assume a lighter colour, viz., from Hochmuth, near Geyer, Saxony. The mineral after treatment in the matrass behaves like sphalerite.

Wurtzite B. B. behaves like sphalerite.

THE COMPOUND OF SULPHIDE WITH OXIDE OF ZINC.

Voltzite behaves like sphalerite, containing only traces of iron.

OXIDE OF ZINC.

Zincite (*red-oxide of zinc*) is infusible. With borax in O. F. dissolves easily and shows manganese. The strongly saturated glass treated a short time in R. F. loses the manganese colour and generally shows a yellow, or bottle-green, iron colour. The glass on coal in R. F. affords a zinc coat. Treated on coal alone, or with soda, it affords a strong zinc coat; on platinum foil a manganese reaction.

OXIDE OF ZINC COMBINED WITH ACIDS.

Goslarite alone in the matrass yields water; with charcoal dust, sulphurous acid. With the fluxes behaves like oxide of zinc, and sometimes shows reactions for manganese, iron, and copper. It is decomposed with soda on coal, yielding a strong zinc coat and sulphide of sodium, which sinks into the coal.

Smithsonite in the matrass yields carbonic acid, and, if nearly free from other metallic oxides, is yellow while hot and white on cooling. The white mass reacts with the fluxes either for zinc alone, or also for iron. If considerable protoxide of iron and manganese are present, the ignited mineral becomes quite dark, is magnetic, and reacts strongly for iron and manganese with the fluxes. Cupriferous smithsonite imparts a lasting green tinge to the flame, even if very little copper is present. The copper can be detected readily with S. Ph. and tin. The unignited mineral dissolves readily with effervescence in the fluxes. Alone, or with soda, on coal in R. F. it is decomposed, and with a sufficiently strong blast may even afford a zinc flame. At first only a cadmium coat is perceptible, but afterward zinc alone.

Hydrozincite yields water in the matrass, loses its carbonic acid, and then behaves like oxide of zinc. Alone on coal in R. F. most of it is gradually volatilized, forming a strong zinc coat, and leaving generally a trifling scoria, which, with borax, shows the iron reactions.

Aurichalcite (*buratite*) yields water in the matrass, and the green (with buratite blue) colour changes to black. Dissolves with effervescence in the fluxes to a clear glass and shows copper. The glass on coal with tin becomes red and opaque on cooling, while a feeble zinc coat is obtained. With soda on coal in R. F. a strong zinc coat and a residue in which metallic copper can be detected by washing.

Iglesiasite fuses in O. F. on platinum foil to a clear yellow glass. With the fluxes it dissolves with effervescence to a yellowish glass, colourless on cooling, which affords a lead coat in R. F. on coal. Alone, or with soda, on coal, it is reduced with effervescence to metallic lead and forms a lead coat, with a second white coat, near the assay, which assumes a green colour with cobalt solution (zinc).

Köttigite in the matrass yields much water. Fuses on coal in O. F. to a globule, which in R. F. evolves an arsenical odour, coats the coal with oxide of zinc, and is black when cold. In the forceps fuses easily to a bead, giving a strong, light-blue flame. Dissolves largely in the fluxes, showing cobalt; the strongly saturated glass on coal in R. F. affords a zinc coat. With soda, or neutral oxalate of potassa, on coal in R. F. much zinc is reduced, forming a very strong zinc coat.

SILICATES OF ZINC.

The *hydrous silicate, calamine* yields water and becomes milk-white in the matrass. B. B. the silicates of zinc are infusible. They dissolve in borax to a clear glass, that cannot be made opaque by flaming; the clear S. Ph. glass becomes cloudy on cooling; and when strongly saturated the still warm glass shows a little separated silica. They are not dissolved by soda alone on coal, but swell and afford a zinc coat with difficulty. With two parts soda and one borax, however, all of the zinc is reduced and volatilized, while the silica fuses with the flux to a glass, which with borax in O. F. sometimes shows iron. Any lead present will cause a slight lead coat behind the coat of oxide of zinc. Silica and the earths present may be found by treating the glass in the wet way. With soda and nitre a manganese reaction is occasionally obtained.

COMPOUNDS OF OXIDE OF ZINC WITH ALUMINA.

Gahnite (*automolite*) is unchanged in the matrass and forceps. Even the fine powder dissolves with extreme difficulty in borax and S. Ph., without showing iron distinctly. It only forms a dark slag with soda, but dissolves readily in a mixture of equal parts of borax and soda, yielding a vitriol-green glass and a distinct zinc coat, if the R. F. is strong enough. With soda and nitre gives a manganese reaction.

To detect alumina and magnesia the glass obtained by treating the mineral with soda and borax is pulverized in the steel mortar and further treated in the wet way.

c. Examination for zinc in furnace products.

The method of testing the products above mentioned, both for zinc and other constituents, may be deduced partly from the general examination for zinc, and partly from the remarks on products containing iron and zinc.

7. CADMIUM, CD.

Its occurrence in the mineral kingdom and in metallurgical products.

Cadmium belongs to the rarer metals and occurs:

a. Combined with *sulphur;*

b. As *oxide* combined with *carbonic acid.*

In metallurgical products cadmium occurs particularly in the oxide of zinc and dust which pass over first during the distillation of zinc from ores.

Examination for Cadmium,

Including the blowpipe characteristics of cadmiferous minerals.

a. General examination for cadmium.

Cadmium can only be detected as oxide by the blowpipe, owing to its volatility. The substance is treated for some time with the R. F. on coal, when the cadmium is volatilized as metal, but oxidizes at once in contact with the air. The oxide is mostly deposited on the coal, and when cold may be recognized by its brown colour, which, however, is only orange-yellow in thin layers. When there is very little cadmium and the coat cannot be easily obtained, the powdered substance should be mixed with soda and treated a very short time in R. F.; a more distinct cadmium coat is then obtained.

In blowing too long some zinc is also volatilized, and its oxide sometimes renders the cadmium coat less distinct.

b. Blowpipe characteristics of cadmium minerals.

Greenockite feebly heated in the closed tube assumes a transient carmine-red colour. In the open tube it yields sulphurous acid. Alone on coal it affords a distinct coat of oxide, and with soda a very abundant reddish-brown coat, while most of the soda sinks into the coal, and evolves an odour of sulphuretted hydrogen when moistened.

For *sphalerite* and *smithsonite*, *vide* zinc.

8. LEAD, PB.

Its occurrence in the mineral kingdom and in metallurgical products.

Lead is quite widely distributed in nature; it occurs:

a. Metallic;
b. Combined with *tellurium*;
c. Combined with *selenium*;
d. Combined with *sulphur*;
e. Combined with *chlorine*;
f. As *oxide*;
g. Combined with *acids*.

Lead occurs under various conditions in metallurgical products obtained in the treatment of plumbiferous ores.

Examination for Lead.

a. Examination for lead in general.

Alloys.

In alloys, as they occur in nature and metallurgical products, lead is recognized by the coat which it affords when the substance is treated on coal in O. F. Any easily volatilizable metals present either pass off entirely in fumes or collect also in a coat as oxides. The oxide of lead coat is dark lemon-yellow while hot and straw-yellow when quite cold, and being also nearer the assay than that formed by some other oxides, as those of tellurium, antimony, and arsenic, is therefore readily distinguishable; it must be remarked, however, that in presence of antimony the oxide of lead coat is darker yellow, like the bismuth coat, probably in consequence of the formation of antimonate of lead (*vide* sulphides, on the following page).

If zinc and lead are present in an alloy, the coat produced is indeed, mingled with oxide of zinc, but the lead' may be recognized by the sulphur-yellow colour of the cold coat as well as by the azure-blue tinge imparted by it to the R. F. When the lead predominates in an alloy of lead and bismuth, the coat appears rather darker than a pure lead coat, but not so dark as a bismuth coat. By testing with S. Ph. the presence of bismuth can be ascertained (*vide* bismuth), provided no antimony is present. Should there be so little lead that it cannot be recognized by the colour of the coat, the latter is treated with the R. F., in order to ascertain whether it disappears with an azure-blue flame, which, in the absence of selenium, indicates lead.

Selenides

Compounds of lead and selenium, with considerable selenium fuse in R. F. with more difficulty than pure lead, and impart an intense azure-blue colour to the flame, chiefly due to selenium, while they emit a seelnium odour and yield at first a slight grayish coat of selenium, followed by a white coat of selenite of lead and a trifling yellow coat of pure oxide of lead.

Sulphides

Combinations of lead with sulphur and other metallic sulphides may be variously examined for lead. The simplest way is to treat a small portion in R. F. on coal, either alone or with a slight amount of borax to separate any iron present, and to recognize the lead by the coat. Unless a large amount of antimony is present, it could not be simultaneously detected with certainty, because adjoining the yellow oxide of lead coat there is formed a white coat of sulphate of lead, which very closely resembles oxide of antimony. It is safer in this case to mingle the powdered substance with a sufficient amount of soda and treat it for a short time in R. F. Sulphide of sodium is formed, and in the absence of antimony only a yellow lead coat with a bluish-white border is produced; but if antimony is present, a white coat of oxide of antimony is obtained beyond the yellow lead coat.

Should the amount of antimony be very trifling, as in many galenas, this method does not afford a perfectly reliable result, because by a prolonged blast some sulphide of sodium is volatilized and also affords a white coat of sulphate of soda. A little antimony can, however, be detected with absolute certainty when present in galenas, and other sulphides consisting mainly of sulphide of lead, by the following process. About fifty milligr. of the powdered substance are placed, with a bit of iron wire as stout as a coarse knitting-needle, in a cylindrical cavity bored in the cross section of a good coal, or in a charcoal crucible, and both are covered with a mixture of soda and borax, the volume of soda being twice, and that of borax once the volume of the substance employed. The whole is then treated in R. F., until all the sulphur is separated, part being combined with the iron and part with the slag.

The lead is nearly all reduced and unites to a button with the antimony, while but little of them is volatilized. The cold lead button is then separated from the slag, and the iron surrounded by sulphide of iron, and is treated with a little soda in R. F. on another coal, when the antimony volatilizes first, producing a coat of oxide, and the lead afterward yields a lead coat. If the antimony coat is touched with the R. F., before a distinct lead coat has formed, it disappears with a greenish-blue flame. The coat of oxide of lead can also be entirely prevented by adding vitrified boracic acid to the antimonial lead, when the acid takes up the oxide of lead and the volatilizing antimony coats the coal with oxide of antimony. This assay is easy, but the following points should be carefully observed. The separation of the sulphur from the lead and antimony must be conducted in a rather deep cavity, so that the antimonial lead may be protected from the air, and as little antimony as possible volatilized. The flame must not be directed immediately upon the separating metallic button, which would then be too strongly heated and might lose antimony by volatilization, but must be directed only upon the slag of soda and borax, which should be made to cover the button. If carefully conducted, quite a trifling amount of antimony may be detected by the coat formed on coal.

When the amount of antimony in a substance containing sulphide of lead is very large, the treatment with soda alone, as well as of the antimonial lead reduced by iron, yields not only an unmistakable antimony coat, but the lead coat is observed to have a darker yellow colour than usual. It appears orange-yellow while hot, and almost lemon-yellow on cooling, quite like a coat of oxide of bismuth, Antimonate of lead seems to be formed, for if this coat is scraped off, dissolved in S. Ph. on platinum wire in O. F., and the bead shaken off and treated with tin on coal, it assumes on cooling a black colour, and becomes quite opaque, which, in the absence of bismuth, indicates antimony.

Lead may also be detected in metallic sulphides by roasting them in fine powder, and then treating them with soda in R. F. Either globules of lead result, or an alloy of lead with other metals, in presence of other easily reducible oxides. A lead coat is likewise formed, to which regard must be had, especially if but little lead is present.

When the substance contains bismuth, as well as lead, it yields by reduction brittle lead, which frequently cannot be recognized as lead at all, when the amount of bismuth is not quite unimportant; the coat on the coal also has a darker colour. In this case the roasted assay must be fused with bisulphate of potassa and further treated as will be directed for such substances under bismuth. The sulphate of lead resulting from the treatment is reduced with soda on coal, and affords metallic lead and a pure lead coat.

If the roasted substance contains much copper, the reduction with soda affords an alloy, in which the lead cannot be recognized by the colour, but after washing the alloy and keeping it fused for some time alone in a strong O. F. on coal, most of the lead volatilizes and affords a coat.

The behaviour of chloride of lead has been given under General Rules, sec. 3. Substances containing oxide of lead with other metallic oxides or earths usually yield a distinct lead coat when treated alone on coal in R. F.; addition of soda, however, renders it more distinct. The same

Oxides.

remark applies to all salts of lead except the phosphate, *which* treated alone on coal fuses to a globule and affords no lead coat, or only a slight one.

b. Blowpipe characteristics of plumbiferous minerals.

Native lead behaves B. B. like pure lead.

Altaite yields in the open tube tellurous fumes, which fuse B. B. to clear drops (G. Rose).

On coal it fuses, tinges the flame greenish-blue and volatilizes, leaving a small silver button.

Clausthalite in the closed tube decrepitates occasionally, but suffers no further change. In the open tube yields a selenium sublimate, red at a distance from the assay and steel-gray nearer to it; a distinct selenium odour is perceptible. On coal it fumes, diffuses a strong selenium odour and fuses only imperfectly in R. F., coating the coal at first with a gray coat of selenium, having a feeble metallic lustre, but appearing reddish further from the assay. A distinct lead coat forms afterward. It gradually volatilizes, leaving a very trifling scoria, which sometimes affords iron, cobalt, or copper reactions with the fluxes.

With soda, or, better still, neutral oxalate of potassa, in R. F. on coal, metallic lead is obtained, which sometimes yields a small silver button on the cupel.

Tilkerodite behaves like clausthalite, but the remaining scoria reacts very strongly for cobalt.

Zorgite.—*Selenkupferblei* behaves like clausthalite, and the remaining scoria reacts very strongly for copper with borax, while with soda it yields a copper button. *Selenbleikupfer* fuses easily, spreads over the coal and forms a gray mass, with a metallic lustre, which after thorough roasting yields a copper button with borax and soda (Berzelius).

Lehrbachite alone in the matrass yields a lustrous, metallic, crystalline, gray sublimate of selenide of mercury, before which a few globules of mercury sometimes collect. With much soda it yields only mercury. In the open tube yields some selenium and a sublimate of selenide of mercury, which fuses to drops. On coal, like clausthalite.

Lead in combination with sulphur and with other metallic sulphides.

Galena and *Bleischweif* usually decrepitate strongly in the closed tube, while a trifling white sublimate is not unfrequently formed, which, however, appears to be only sulphur. In the open tube yield sulphurous acid and at a higher heat a white sublimate of sulphate of lead, gray just above the assay. Fuse with difficulty on coal, until most of the sulphur is expelled, when metallic lead separates. The coal is strongly coated with sulphate and oxide of lead. In presence of antimony the sulphate coat is mingled with oxide of antimony. Iron and zinc in galena are found under iron and zinc. More or less silver may be detected in most galena by cupelling the lead reduced on coal (*vide* quantitative silver assay).

Steinmannite from Przibram decrepitates rather strongly in the closed tube. On coal fuses, evolving sulphurous and antimonous fumes and leaving a lead button, which affords silver when cupelled.

The general behaviour of these minerals is as follows:—

In the closed tube they decrepitate more or less and are distinguished by their fusibility when the tube is treated B. B. Those containing most sulphide of antimony are the most fusible, and they afford a red sublimate of amorphous tersulphide of antimony, containing oxide of antimony. In the open tube they yield antimonial fumes, partly volatile and partly fixed. The former consist of oxide of antimony; the latter partly of oxide with antimonic acid, partly of sulphate of lead, and partly of antimonate of lead, near the assay. Much sulphurous acid is also evolved.

On coal they fuse and deposit thick coats; the farthest removed are white and consist of oxide of antimony with sulphate of lead, while near the assay the coat is chiefly oxide of lead, but dark yellow and mixed with antimonate of lead. Any trifling amount of iron and copper usually remains as a scoria and can be tested after expelling the Pb and Sb.

With soda in R. F. they are decomposed and yield metallic globules and coats of lead and antimony. To determine whether the white coat consists only of Sb, or whether it also contains sulphate of lead (*vide* p. 236). Arsenic, if not in too small quantity, can frequently be detected in the tubes and on coal by its characteristic behaviour.

Dufrenoysite contains the Pb combined with As alone; in the closed tube yields a red sublimate of sulphide of arsenic; in the open tube a smell of sulphur only, with a sublimate of sulphur in the upper part of the tube and of arsenous acid below. (Dana.)

On charcoal fuses very easily, evolves sulphurous and arsenous fumes, and finally yields a lead button.

Clayite.—As regards the blowpipe characteristics it is only known that it fuses readily, yields reactions for lead, arsenic, and antimony, and leaves with soda a lustrous metallic globule; no doubt after treatment with boracic acid (*vide* copper assay), the remaining button would afford copper reactions.

Cuproplumbite. B. B. in the open tube swells up and fuses, affording sulphurous acid and a trifling sublimate of sulphate of lead. Alone on coal in R. F. yields coats of oxide and sulphate of lead; with soda a button of metal, somewhat harder than pure lead, which after treatment with boracic acid (*vide* copper), leaves a copper button. This cupelled with test lead affords some silver also.

Alisonite; like cuproplumbite.

Nagyagite.—The antimonial variety strongly heated in the open tube fumes and deposits a coat, which is gray close above the assay and seems to consist of tellurate, antimonate, and perhaps sulphate of lead. The coat further removed consists partly of volatile oxide of antimony, and partly of tellurous acid, which can be fused to clear drops.

Alone on coal it fumes and deposits a white, volatile coat of mingled oxide of antimony, tellurous acid and sulphate of lead, and a less volatile, yellow coat, consisting chiefly of oxide of lead, with perhaps some antimonate. The first coat disappears under the R. F. with a bluish-green, the latter with a blue flame. When all the volatile ingredients are expelled, a malleable gold button remains, which assumes a pure gold colour when cupelled with test lead.

If the flame test does not show tellurous acid distinctly in the white coat on coal, it is only necessary to treat the powdered mineral with boracic acid, when a coat of oxide of antimony and tellurous acid will be obtained, which will disappear with a bluish-green flame in R. F. The behaviour of the variety free from antimony is similar, but the tellurium reactions are more distinct.

Bournonite decrepitates in the closed tube and strongly heated yields a slight dark-red sublimate of sulphide with oxide of antimony. In the open tube evolves sulphurous acid and copious white, antimony fumes. The coat condensing on the upper side of the tube is volatile oxide of antimony; that on the lower part is a non-volatile compound of antimonate of antimony with considerable antimonate of lead.

Alone on coal fuses very easily, affording a coat of oxide of antimony, quickly followed by $\dot{\mathrm{Pb}}$, $\dddot{\mathrm{Sb}}$, $\dot{\mathrm{Pb}}$, S, and $\dot{\mathrm{Pb}}$, so that nearest the assay the coat is dark-yellow. Finally only a lead coat is afforded. The remaining globule tested with borax or S. Ph. shows copper and sometimes antimony. It is rendered somewhat brittle by sulphur and antimony, and after cupellation occasionally affords a little silver.

Kobellite fused in the closed tube B. B. yields a slight sublimate of sulphur. In the open tube yields copious antimonial fumes and sulphurous acid; it is not fused, but is covered with yellow oxide. On coal fuses easily, yielding a white coat of oxide of antimony with sulphates of lead and bismuth, and a yellow coat, which is dark pomegranate-yellow on cooling and disappears with a feeble blue flame in R. F. Small, somewhat malleable, metallic buttons remain, which, after being fused together for some time in O. F., show copper, with S. Ph. (*vide* copper).

Freed from most of its sulphide of antimony on coal in O. F., the mineral shows iron when treated with borax in R. F. and the glass remelted in O. F. on platinum wire. The bismuth is found by roasting the mineral on coal, and fusing the fine powder with three or four volumes of bisulphate of potassa (*vide* bismuth).

Aikinite in the open tube yields white fumes, partly fusible to clear drops, white on cooling; sulphurous acid is evolved.

The remaining button is surrounded by black, fused oxide, which is transparent and greenish-yellow on cooling.

On coal fuses, fumes and deposits a white coat, the inner edge of which is yellow. The resulting button resembles bismuth (Berzelius). With the fluxes gives copper reactions and after long blowing a globule of metallic copper (Dana).

LEAD COMBINED WITH CHLORINE.

Cotunnite fuses to a yellow fluid and partially sublimes in the matrass. On coal fuses very easily, spreads out and volatilizes, forming a white coat of chloride of lead. This disappears with an azure-blue flame in R. F. and leaves a yellow spot of oxide of lead. With soda yields lead and with oxide of copper affords the chlorine reaction (*vide* chlorine).

Mendipite decrepitates, becomes yellow, and when strongly heated behaves like cotunnite. On coal fuses easily, emits acid vapours and is reduced to metal, affording also a white coat of chloride and a yellow coat of oxide of lead. A special test shows chlorine.

Matlockite.—Like mendipite.

Phosgenite behaves like mendipite, but effervesces with nitric acid.

OXIDES OF LEAD.

Massicot (*plumbic ochre*), *minium*, and *plattnerite* behave quite like oxide of lead.

OXIDE OF LEAD COMBINED WITH ACIDS.

Anglesite decrepitates in the matrass and usually yields a little water. On coal in O. F. fuses to a clear bead, opaque on cooling, and in R. F. is reduced with effervescence to a lead button. Reduced with soda yields lead, and a strong sulphur reaction is afforded by cutting out the coal and laying it on moistened silver foil. Trifling admixtures of oxide of iron or manganese may be easily detected by the tests with borax on soda and nitre.

Linarite yields some water and loses its blue colour in the matrass. On coal fuses in O. F. to a bead; in R. F. is reduced with effervescence to lead, which then affords a lead coat. The lead button treated with boracic acid yields a button of copper. Reduced with soda it yields copper and lead, while the alkaline mass that sinks into the coal gives a sulphur reaction.

Caledonite.—B. B. on coal easily reduced. Partially soluble with effervescence in nitric acid, leaving a residue of sulphate of lead. (Dana.)

The copper and also the sulphuric acid may be detected as in linarite above.

Lanarkite.—Like caledonite, but no copper. In O. F. fuses to bead, white on cooling and containing reduced lead.

Leadhillite swells a little on coal in a feeble flame, and assumes a transient yellow colour; in a stronger flame is easily reduced.

Effervesces with nitric acid and leaves a residue of Pb S.

Pyromorphite sometimes decrepitates in the matrass, and strongly heated affords a trifling, volatile sublimate of Pb Cl. In the forceps fuses very easily to a globule and affords a blue flame with a green tip (phosphoric acid), especially when the blast is gentle. The fused portion has a crystalline surface.

On coal at first yields a slight coat of Pb Cl and the fused button is not reduced, but on cooling glows again and shows quite lustrous facets. The Pb Cl coat meanwhile increases, and a pale yellow oxide of lead coat can be seen around the assay. If, however, arsenate of lead is present, it is reduced with effervescence and emission of arsenical fumes to lead, which remains with the crystalline phosphate. The powdered mineral fused with oxide of copper on coal gives an azure-blue chloride of copper flame.

By reduction with soda it affords lead, which sometimes yields a trace of silver when cupelled. Fused in the platinum spoon with three to four parts of bisulphate of potassa, it forms a clear mass, white on cooling. (Vanadate of lead affords a pomegranate-yellow mass, and chromate of lead a mass, which is violet while hot and greenish-white on cooling.)

Mimetite fuses on coal somewhat less easily than pyromorphite, but is then reduced, with effervescence and a strong arsenic odour, to lead. At first affords only a chloride of lead coat, but later arsenous acid and oxide of lead. The presence of phosphate causes the appearance of one or more of the crystalline beads above mentioned. Chlorine is detected as under pyromorphite. Lime is found by decomposing the mineral with soda. The lead separates as metal, part of the soda sinks into the coal, and part forms with the lime an infusible mass.

Plumbogummite decrepitates and yields much water in the matrass. Tested afterwards in the forceps, it swells like a zeolite, and colours the flame azure-blue, but fuses only imperfectly. On coal cannot be melted, but gives a faint white coat of Pb Cl.

In the fluxes dissolves easily to a clear glass; the S. Ph. bead is opaque with a large addition. With soda yields lead globules and with cobalt solution turns blue. With boracic acid and iron gives a phosphide of iron (*vide* phosphoric acid).

Cerussite decrepitates in the matrass, loses carbonic acid and turns yellow, with a stronger heat dark-red, but the yellow colour returns on cooling.

Alone on coal is easily reduced; in the fluxes it dissolves with effervescence and gives the reactions of oxide of lead. Dilute nitric acid also dissolves it with effervescence.

Earthy carbonate of lead. Like cerussite, but leaves on reduction a trifling scoria, which reacts for iron with borax.

Phœnicochroite (*melanochroite*) fuses readily in O. F. to a dark mass, assuming a crystalline structure on cooling. In R. F. gives off lead fumes and is decomposed into sesquioxide of chromium and metallic lead. With the fluxes gives chromium reactions.

Crocoite decrepitates and flies into small bits, assuming transiently a darker colour. On coal fuses, spreads out, and is suddenly reduced with deflagration to lead, affording also a lead coat, while grayish-green sesquioxide of chromium remains with the lead.

Dissolves easily in the fluxes in O. F. to yellowish glasses, becoming green on cooling. In R. F. the green is darker.

Reduced with soda yields lead, and when fused on platinum foil with soda affords a dark-yellow mass, becoming light-yellow when cold. Fused in the platinum spoon with three to four parts of bisulphate of potassa, a quite dark-violet mass results, which is reddish on solidifying and when cold is greenish-white. (Vanadate of lead imparts a yellow colour to the salt.)

Vauquelinite. B. B. on coal swells a little and then fuses with frothing to a gray, lustrous, metallic globule, showing reduced buttons of metal where it is in contact with the coal; a distinct lead coat is formed.

With the fluxes gives in O. F. green beads, both hot and cold, but in R. F. they become on cooling red, opaque red, or nearly black, according to the amount dissolved. With tin the red copper colour is more distinct.

Dissolves with effervescence in soda on platinum wire in O. F., giving a clear green glass, yellow and opaque on cooling. This forms a yellow solution with water, in which chromic acid can be detected (*vide* chromium).

It is completely reduced with soda on coal, and by treating the lead buttons, after washing away the coal, etc., with boracic acid, copper is obtained (*vide* copper).

Descloizite in the matrass yields some water. B. B. fuses, is partially reduced to lead, surrounded by a black slag, and gives a lead coat. With borax in R. F. a green glass; in O. F., on adding nitre, a violet glass (manganese). With S. Ph. in O. F. a yellow, in R. F. a chrome-green glass.

Dechenite fuses easily to a yellow glass in the forceps, and also in the matrass, without decrepitating. On coal fuses readily to a yellowish-green bead, and gives the usual lead globules and coat. In several assays a distinct arsenical odour was observed, but not in others made with pure, transparent fragments. Phosphoric acid cannot be detected. The glass fluxes afford only vanadic acid reactions. Soda causes a white enamel, showing lead globules. (Bergmann.)

Vanadinite from Zimapan decrepitates in the matrass, and at a high heat gives a trifling white sublimate.

On coal in O. F. the powder fuses easily to a black, somewhat lustrous mass, which yields lead in R. F. At first a slight Pb Cl coat is formed, and later one of oxide of lead. After driving off the lead, the dark-gray residue gives with S. Ph. in R. F. an emerald-green bead.

Dissolves readily in the glass fluxes, showing vanadic acid reactions. With soda in O. F. on platinum wire fuses to a yellow mass, crystalline and lighter in colour when cold. On coal a lead button separates. Chlorine is detected by the S. Ph. bead containing oxide of copper.

Fused in the platinum spoon with three to four parts of bisulphate of potassa, it forms a clear, yellow, fluid salt, assuming a red and finally a pomegranate-yellow colour on cooling; thus differing at once from crocoite and pyromorphite.

Chileite fuses in the candle-flame to a black bead. In S. Ph. gives a green glass, and with soda on coal a cupriferous lead button. (Domeyko.)

Wulfenite decrepitates in the matrass and transiently assumes a darker colour. On coal fuses and sinks partly into the coal, while lead separates and a lead coat is formed. By long treatment with R. F. most of the lead is volatilized, and then by washing the portion which has sunk into the coal, a mixture of malleable lead and an alloy of lead and molybdenum.

Dissolves readily in borax in O. F. to a clear yellow bead, colourless on cooling, which becomes opaque black in R. F. This, if pressed flat, appears dirty-green, with black flocks of binoxide of molybdenum, especially on coal. In S. Ph. on wire dissolves readily to a yellowish-green glass, losing much of its colour on cooling, and becoming dark-green in R. F. With soda on coal affords lead.

When fused with bisulphate of potassa in the platinum spoon, it forms a yellowish mass, becoming white on cooling and this, dissolved by treating it in distilled water, affords a solution which very quickly becomes dark-blue on adding a bit of zinc or tin.

Stolzite decrepitates in the matrass. On coal fuses, with formation of a lead coat, to a globule that crystallizes in cooling and has a dark metallic surface, while it shows a grayish-white, vitreous fracture. Dissolves easily in the glass fluxes in O. F. to a clear colourless bead. (The borax bead in R. F. becomes yellowish, and sometimes on cooling is gray and opaque.) The S. Ph. bead in R. F. after short treatment assumes a blue colour, but sometimes not so pure as from tungstic acid alone. Too large a quantity, or too long blowing, renders the glass greenish and finally quite opaque. Reduced with soda yields lead. With soda and nitre a distinct manganese reaction. Fused like the preceding mineral with bisulphate of potassa and dissolved, the solution gradually becomes grayish-blue on adding zinc or tin. To detect tungstic acid readily in the wet way, see the examination for tungstic acid.

Bindheimite yields water, and becomes darker in the matrass. On coal is reduced to a metallic button, which volatilizes gradually, coating the coal with oxides of lead and antimony.

c. *Examination for lead in metallurgical products, together with their blowpipe characteristics.*

The various samples of commercial lead sometimes contain more or less copper, antimony, and arsenic. If such lead is fused and kept in rotary motion, B. B. on coal, arsenic can be detected by the odour, and antimony by the coat deposited beside the lead coat. A very trifling amount of antimony can be found by using boracic acid (*vide* antimony).*

To detect any copper present, a bit of the lead is treated with vitrified boracic acid on coal, until nearly all of the lead is slagged off, when the remaining metal button is fused with S. Ph. in O. F. (*vide* copper).

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* To the practical smelter the blowpipe may be very especially recommended as furnishing an easy and certain means of recognizing both of the above-mentioned ingredients in lead. Even very trifling quantities of arsenic can be immediately detected by the odour, when about two grm. of the lead are quickly fused in a cavity on coal with the blue flame, and kept in rotary motion for a few moments. A little antimony, if present, may be recognized by the bluish-white coat of oxide of antimony, which forms before the yellow lead coat appears. In any case the most minute admixture of both these ingredients is at once perceptible, because upon cooling the lead button has not the characteristic lead-gray, lustrous surface, covered with glistening yellowish litharge, which is shown by quite pure lead, but presents a dull, blackish-gray colour.

Silver is found by cupelling the lead (*vide* silver assay). Plumbiferous *black copper* varies in character, but always yields a distinct lead coat. Other ingredients are found according to the directions under the general examination for iron in alloys. The behaviour of *speisses* and *matts* is also given under iron.

Litharge behaves B. B. like oxide of lead. If containing arsenate or antimonate of lead, it affords, on reduction, an arsenic odour, or an antimony coat. Copper is found by reducing some litharge to metallic lead and treating the button with boracic acid and afterward with S. Ph., using tin if necessary (*vide* copper).

Abstrich from the cupellation for silver when treated alone on coal is reduced to metallic lead, emits a strong arsenic odour and yields coats of lead and antimony. Copper is found as in litharge. To detect iron a larger piece of abstrich is reduced beside a small borax bead on coal, keeping the glass constantly covered with a good R. F., and shaking off the reduced lead upon the anvil from time to time. By remelting this glass on platinum wire in O. F. the iron can be raised to the state of sesquioxide and recognized.

To detect sulphuric acid, not too small a quantity of the abstrich is powdered and reduced with soda on coal, the resulting fused mass cut out from the coal and laid on moistened silver foil (*vide* sulphuric acid).

Abzug behaves in general quite like abstrich, but if much of the hearth mass is mixed with it, the metallic oxides can only be reduced by adding borax, or soda and borax.

Cupel bottoms or *hearths*, from the cupellation for silver, as well as the *flue rakings* and *lead smoke* from lead-smelting, cupelling and roasting processes, at once yield a lead coat when treated alone on coal in R. F. The manner of detecting the other ingredients may be inferred from what has preceded.

Slags containing oxide of lead yield a lead coat either alone on coal in R. F., or when fused to a globule with soda. The other ingredients are found by the method given under lime.

9. Tin, Sn.

Its occurrence in the mineral kingdom and in metallurgical products.

Tin occurs, in nature, in the following minerals:—

a. Combined with *sulphur;*

b. As *oxide.*

Tin is likewise found as an unessential ingredient in several other minerals, viz., *meteoric iron, titanic iron, tantalite,* &c. Traces of tin can also be detected in certain brown and black zinc blendes, when quantities of several grammes are treated in the wet way.

This metal is rarely to be sought for among metallurgical products except in the especial products of the tin-works, including:—

a. The varieties of *tin* in commerce;

b. The *scraps* obtained in smelting tin ores and refining tin;

c. Deposits which form on the soles of the shaft furnaces when smelting tin ores;

d. Tin slags.

When copper ores occur with stannite or cassiterite and cannot be perfectly separated in extracting and dressing the ores, a stanniferous copper matt is formed during the smelting process, while stanniferous copper is especially separated during the fusions for concentration.

Examination for Tin.

a. General examination for tin.

The behaviour of metallic tin and the coat of oxide on charcoal have been given on p. 67. If the tin contains lead or bismuth, it is scarcely possible to keep a globule of the alloy in fusion, even in the best R. F., without having it covered with a crust of oxide. By adding borax, however, and treating this with the R. F., an admixture of lead or bismuth may be recognized by the yellow coat deposited on the coal. Should it be doubtful whether lead or bismuth is present, the coat is carefully scraped off, dissolved in S. Ph. on platinum wire, and the bead treated on coal with tin. Bismuth is indicated by the gray or black appearance of the cold bead (see tables). The presence of arsenic is also ascertained by the odour, while treating the alloy with the borax, which afterwards oxidized on platinum wire will also show a yellow colour in presence of iron. Tin is tested for copper by fusing it on coal with a mixture of one hundred parts by weight of soda, fifty borax, and thirty silica, as described in the separation of tin from copper under the quantitative copper assay. The remaining button of copper, containing only a little tin, is treated with S. Ph. on coal in O. F., until the glass is coloured. The S. Ph. glass may then be further tested with tin, to ascertain whether copper is actually present or not. Tungsten, which usually is present only in trifling quantities, cannot always be detected with certainty by the fluxes, because the tin is seldom quite free from iron; but on dissolving enough of the tin by warming it with aqua regia, diluting with water, decanting the clear solution after the residue has settled, and digesting the latter with fresh aqua regia, yellowish-green tungstic acid remains, if the tin contained tungsten. The acid solution, usually yellow from iron, is again decanted, the residue of tungstic acid washed with water, and tested with S. Ph. on platinum wire, when after a short treatment in R. F. it affords a blue bead.

Tin, when present in alloys, is almost always detected on fusing them upon coal, since the globule cannot be kept bright even in the R. F., but quickly becomes covered with an increasing crust of oxide, which can only be removed with difficulty after adding borax.

Sulphides.

Metallic sulphides containing tin, but yielding no coat of oxide of tin near the assay when treated alone on coal, must be roasted and treated in R. F. with soda and borax, when metallic tin is obtained, which may be tested alone on coal. If other reducible metals are present, they afford an alloy, in which the other metals can be recognized by means of the fluxes.

Metallic oxides.

In metallic oxides, or substances generally, which are composed of oxides, tin may be best detected by a reduction assay on coal with soda, or neutral oxalate of potassa; it is, however, necessary, in certain cases, to add borax, so as to slag off the considerable amount of iron present.

b. Blowpipe characteristics of tin minerals.

Stannite alone on coal in R. F. fuses to a globule. In O. F. it evolves sulphurous acid and is covered with oxide, which is also deposited on the coal near the assay and can be immediately recognized by its well-known properties.

In the open tube it yields sulphurous acid and some oxide, which collects quite near the assay and cannot be volatilized again. Well roasted with alternate O. F. and R. F., it shows iron and copper with borax.

A little zinc, which may be found in the wet way, cannot be detected B. B., since the coat which it forms is concealed by the oxide of tin.

Cassiterite behaves like binoxide of tin (see tables), but with a sufficient quantity the borax bead occasionally shows a yellowish colour, only while hot, owing to some iron; a feeble manganese reaction is frequently obtained with soda and nitre. A small quantity of tantalic or hyponiobic acid can be found by the method to be given under tantalum and niobium.

Stannite is infusible on coal and in the forceps. It dissolves slowly to a colourless glass in borax and S. Ph. giving a silica skeleton with the latter. With little soda fuses to a slag-like mass, with more in R. F. affords metallic tin.

c. Examination for tin in metallurgical products, including their blowpipe characteristics.

The behaviour of commercial *tin* may be deduced from the remarks on tin in general.

The different sorts of *tin scraps* from smelting and refining tin behave variously, but their constituents may be quickly detected by testing them on coal and with the fluxes, observing all that was said under *a.*

The deposits on the sole of the furnace, treated on coal with borax in R. F., sometimes yield an unmistakable coat of oxide of tin. The manner of detecting the remaining ingredients may be deduced from the general remarks on metallic compounds under iron.

Should there be so little tin, however, as to yield no distinct coat, it is only necessary to dissolve the product in nitric acid and test the residue of binoxide of tin with soda on coal.

Tin slags fuse in R. F. alone on coal, without producing a noticeable coat, but by a reduction assay with soda and borax metallic tin is obtained. The other ingredients are detected as given under lime, but some silver must be added when it is proposed to reduce all the oxide of tin. If the slag contains tungstic acid, the whole of the tungsten is found in the separated silica (*vide* tin slags, under tungsten).

10. BISMUTH, Bi.

Its occurrence in the mineral kingdom and in metallurgical products.

Bismuth belongs to the rarer metals; it is found:

a. *Metallic;*
b. Combined with other *metals;*
c. With *sulphur* and *arsenic;*
d. With *sulphur;*
e. As *sulphide* combined with *oxide;*
f. As *oxide;*
g. Combined with *carbonic acid;*
h. With *telluric acid;*
i. As *silicate* with *phosphate.*

Bismuth, which is generally extracted from cobalt ores by a simple liquation process, also forms an occasional accessory ingredient in certain metallurgical products; viz. as metal, in larger or smaller proportion, in *cobalt speiss*, when the cobalt ores employed in the production of smalt contain bismuth. Bismuth is also concentrated in the *speiss* and *lead matt* obtained as intermediate products in smelting cobalt ores containing bismuth and silver; it is frequently present in "brightened" silver, and most of it is absorbed by the mass of the hearth when the silver is refined. Lastly, a little bismuth may occur in tin, produced on a large scale, unless the roasted tin ore has been freed from it by treatment with dilute hydrochloric acid, etc., previous to the smelting process.

Examination for Bismuth.

a. General examination for bismuth.

Alloys. Bismuth in alloys, as they occur in nature and among metallurgical products, may be recognized by the coat afforded by the substance alone on coal, and which is best obtained in R. F. It is dark orange-yellow while hot, lemon-yellow on cooling, and changes its place under the R. F., without colouring the flame. When easily volatilizable metals are present, they partly pass off, partly in fumes and partly afford a coat, adjoining the bismuth coat, *e. g.*, tellurium, arsenic, etc.

Bismuthiferous lead, which is more or less brittle according to the proportion of bismuth, is treated alone on coal, until a distinct coat is produced, which is carefully scraped off, dissolved in S. Ph. on platinum wire in O. F., and the colourless bead treated with tin on coal in R. F. If bismuth was present, the cold bead is dark-gray or nearly black. Acids of antimony, however, produce a similar reaction, and the metallic compound must first be freed from antimony, if present, by treating it for some time on coal in O. F., and then keeping it melted on a fresh coal, until a coat is formed, which will suffice for the S. Ph. test.

To very infusible alloys, containing nickel, for instance, some pure silver must be added, and the whole treated in R. F.

Sulphides. When the bismuth is combined with sulphur, a white coat of sulphate of bismuth forms beyond the yellow coat, but this may be prevented by adding soda.

The presence of much lead causes a mixed coat of oxides of lead and bismuth, which can hardly be distinguished from a pure lead coat, and a trifling amount of bismuth can then only be detected by a special test, which may be made in two ways. The simplest method consists in producing a copious yellow coat and testing it as before with S. Ph., after first ascertaining the absence of antimony by a special examination for that metal.

The other method consists in roasting the substance thoroughly, but carefully, on coal, to prevent *sintering,* fusing it then with three to four volumes of bisulphate of potassa in the platinum spoon, and treating the mass with water in a small porcelain dish over the lamp, until everything is detached from the spoon. Sulphate of potassa and other soluble sulphates are dissolved, leaving neutral sulphate of lead and basic sulphate of bismuth; only a trifling quantity of the bismuth is dissolved as neutral sulphate. Antimony if present, also remains behind as acid.

After decanting the clear solution, the residue is heated to boiling with pure water, a few drops of sulphuric acid and some nitric acid, when the sulphate of bismuth dissolves, leaving a residue of sulphate of lead, with any oxide of antimony present. After filtration the bismuth is thrown down from the warm filtrate by means of S. Ph., as a white precipitate, which is collected on a filter and tested with S. Ph. The bead on platinum wire is colourless, or only yellowish, but on coal with tin in R. F. becomes dark-gray on cooling, behaving therefore like oxide of bismuth. It may also be recognized as such on coal.

Oxides, etc.

When substances containing oxide of bismuth are treated alone, or with soda, on coal, they yield an unmistakable bismuth coat. Should there be any doubt, the coat may be scraped off and tested with S. Ph., provided the substances are free from oxide of antimony.

b. Blowpipe characteristics of minerals containing bismuth.

Native bismuth behaves like pure bismuth (see General Rules).

COMPOUNDS OF BISMUTH WITH TELLURIUM, SULPHUR, AND SELENIUM, SEPARATELY AND TOGETHER.

Tetradymite, free from sulphur, fuses easily and yields in the open tube white fumes, which partly stream through the tube and partly condense near the assay; when selenium is present, a strong heat causes a red spot, due to the admixture of selenium, which also imparts a strong odour of selenium to the escaping gas. The white coat fuses B. B. to clear, colourless drops, and is thus recognized as tellurous acid, but the reddish film volatilizes. After the volatile constituents are mostly expelled, the metallic globule is surrounded by brown fused oxide of bismuth, which is opaque and yellow when cold. On coal fuses very easily to a metallic globule, colours the flame bluish-green, diffuses, if seleniferous, a distinct selenium odour, and deposites a white coat, with a dark orange-yellow one still nearer the assay. The former disappears under the R.F. with a bluish-green flame, the latter becomes lemon-yellow on cooling. The remaining button can be entirely volatilized, furnishing an abundant bismuth coat.

The sulphurous varieties also yield sulphurous acid in the tube.

BISMUTH WITH ARSENIC AND SULPHUR.

Alloclasite affords arsenic fumes and a bismuth coat on coal, and fuses to a dull gray button (Naumann).

BISMUTH COMBINED WITH SULPHUR AND OTHER METALLIC SULPHIDES.

Bismuthinite fuses in the closed tube, yielding a little sulphur. Carefully heated in the open tube it fuses, yielding sulphurous acid and a coat of sulphate of bismuth, which fuses B. B. to brown drops, yellowish and opaque on cooling. Strongly heated, the assay boils, and oxide of bismuth is deposited in the surrounding glass. On coal it first yields some sulphur, then fuses, spirts out glowing drops, and deposits coats of oxide and sulphate of bismuth. When all the bismuth is removed, a trifling scoria usually remains, which frequently affords iron reactions and sometimes copper with the fluxes.

SULPHIDE WITH OXIDE OF BISMUTH.

Karelinite yields some sulphurous acid, but no sulphur, in the matrass; metallic globules of bismuth separate from the fused mass. In the open tube sulphurous acid and a button of metal, surrounded by easily fusible oxide of bismuth. (Hermann.)

OXIDE OF BISMUTH.

Bismite generally yields water, and upon addition of hydrochloric acid frequently shows a little carbonic acid. With fluxes and on coal, like oxide of bismuth.

SALTS OF BISMUTH.

Bismutite in the matrass decrepitates, yields water, becomes brown, and fuses readily on the glass. On coal is quickly reduced to bismuth. In S. Ph. a dark-yellow bead, colourless on cooling and showing flakes of silica. Dissolves in nitric acid with effervescence, leaving a yellow clayey residue. (Rammelsberg.)

Carbonate of bismuth (*Bismuthit*) yields in the matrass a little water, decrepitates and turns gray. On coal reduces with effervescence to bismuth, which affords a bismuth coat and can be driven away, leaving a trifling scoria. This in R. F. fuses to a magnetic globule, and shows chiefly iron and copper with the fluxes. With soda on coal the powdered mineral affords a sulphur reaction. It dissolves in hydrochloric acid, with evolution of carbonic acid to a pale, yellowish fluid.

Montanite, according to Dana, yields water in the closed tube. B. B. gives reactions for bismuth and tellurium.

Eulytite is unaltered in the closed tube. (According to Dana, decrepitates and affords a trace of water.) In the forceps fuses very readily with intumescence, and if pure, tinges the flame bluish-green (phosphoric acid). On coal swells and fuses easily to a brown bead, deposits a bismuth coat and sometimes emits an arsenical odour. With a little soda effervesces and yields metallic bismuth and a bead, coloured blue by a little cobalt. If this bead is powdered, treated with acetic acid, diluted with water, filtered, and the solution treated with a few drops of solution of acetate of lead, phosphate of lead forms, and produces a distinct cloudiness. The S. Ph. glass shows silica, and with tin on coal bismuth.

Hypochlorite is infusible in the forceps, but assumes a dark-brown colour. With sulphuric acid the powder yields a distinct phosphoric acid flame. On coal in R. F. yields a trifling bismuth coat, without fusing. Dissolves slowly in borax, showing iron, and the saturated glass in R. F. on coal becomes somewhat cloudy and then assumes a yellowish-green colour, while a slight bismuth coat is formed. The S. Ph. bead is yellow and shows silica; on coal with tin becomes dark-gray.

With soda on coal it fuses with effervescence to a globule, and produces a distinct bismuth coat, while a slight manganese reaction is obtained with soda and nitre.

c. *Metallurgical products.*

The remarks under the general examination for bismuth apply here.

11. URANIUM, U.

Its occurrence in the mineral kingdom.

Uranium is found:

a. As *oxide;*

b. Combined with acids.

Examination for Uranium.

a. General examination for uranium.

When testing for uranium the chief point to be considered is the behaviour of its sesquioxide with S. Ph., with which in O. F. it yields a yellow glass, becoming yellowish-green on cooling and pure green in R. F.

In absence of other oxides producing similar colours, S. Ph. yields decisive results, but when oxides of iron and possibly also titanic acid are present, in which case the S. Ph. bead in R. F. becomes red on cooling (*vide* iron), the uranium colour can only be perceived by treating the glass in O. F., when it assumes on cooling a green colour, mixed with much yellow.

When there is little uranium and much iron the fluxes show only the iron, and the substance must then be treated with bisulphate of potassa, carbonate of ammonia, etc. (*vide* iron).

Substances containing oxides of copper and uranium yield green beads in O. F. with borax and S. Ph., and as substances containing oxides of iron and copper, without uranium, do the same, the following method may be adopted to detect the presence of a little uranium. The substance is treated with soda, borax, and a silver button on coal in R. F., until all the copper is reduced into the silver, after which the slag, containing uranium and other non-reducible oxides like oxide of iron, in a low state of oxidation, is dissolved by warming it with a little aqua regia, treated with excess of carbonate of ammonia, and the process conducted according to examination for iron.

b. Blowpipe characteristics of uranium minerals.

Uraninite from Johann-Georgenstadt yields some water at first, then usually, if containing many foreign substances, a trifling sublimate of sulphur, next sulphide of arsenic, and finally metallic arsenic.

In the open tube evolves sulphurous acid, and a ring of arsenous acid collects on the tube: the assay does not alter perceptibly. B. B. is only rounded somewhat on the edges and usually tinges the flame azure-blue (lead), near the assay, and fine green at a greater distance (copper).

The thoroughly ignited mineral behaves with the glass fluxes like sesquioxide of uranium (see tables).

It is not dissolved by soda, but if treated in R. F. on coal, which often causes a perceptible odour of arsenic, after washing away the coaly particles and protoxide of uranium in the mortar, it yields metallic particles of a light copper-colour, apparently consisting of plumbiferous copper, since a yellow coat is also produced on the coal. A test with S. Ph. establishes this supposition.

Coracite, gummite, eliasite, yield much water; otherwise like uraninite.

The *sulphates of sesquioxide of uranium* yield water in the matrass, become red, then brown; on coal evolve sulphurous acid, and react for uranium with the fluxes.

The *sulphates of the proto-sesquioxide* behave similarly, but B. B. change to a brown or greenish-black mass. The S. Ph. bead with tin on coal is dark-red in presence of copper. The nitric acid solution treated with water and ammonia in excess yields a yellow precipitate, which behaves like pure sesquioxide of uranium with the fluxes. If Cu is present, the ammoniacal solution is blue.

With soda on coal they all yield a strong sulphur reaction.

Autunite yields water and becomes opaque straw-yellow in the matrass. On coal fuses with some intumescence to a black globule, with a crystalline surface. With the glass fluxes shows uranium; with soda a yellow, unfused slag. (Berzelius.)

Torbernite behaves like autunite, but shows copper with S. Ph. and tin, as well as on reduction with soda. The copper button is frequently whitened by arsenic, which can be recognized B. B. by its odour.

Liebigite yields water and becomes greenish-gray in the matrass. At a red-heat blackens, without fusing, but becomes orange-red on cooling.

B. B. in forceps and on coal is infusible, but remains black.

With borax in O. F. a yellow bead, green in R. F. Dissolves with lively effervescence in hydrochloric acid, forming a yellow solution. (J. L. Smith.)

The related *Uran-Kalkcarbonat* loses water and becomes grayish-black, or by access of air brownish-black, is infusible, and shows uranium with fluxes.

Voglite likewise blackens when heated, is infusible and colours the flame green. It yields copper when reduced with soda.

Uranophane yields alkaline water in the matrass, blackens, and on cooling is rusty brown. In the open tube becomes almost orange-yellow, and strongly heated yields trifling vapours and a coat, partly volatile and partly fusible to drops, Te, while a feeble selenium odour is perceptible. Alone the mineral fuses to a black glass and imparts a slight copper coloration to the flame. On coal affords coats of Sb and Bi. With the fluxes shows Si and uranium.

12. COPPER, Cu.

Its occurrence in the mineral kingdom and in metallurgical products.

Copper occurs very frequently in nature, being found:
a. Metallic;
b. Combined with *arsenic;*
c. Combined with *selenium;*
d. Combined with *sulphur;*
e. Combined with *chlorine;*
f. As *oxide;*
g. Combined with *acids.*

Copper is also frequently found, in addition to the regular copper products, as an accessory ingredient of the silver and lead products of smelting-works, when the ores treated were cupriferous.

Examination for Copper.

a. General examination for copper.

Alloys.

Most native combinations of copper with other metals contain selenium; when this is expelled in O. F. on coal, other easily volatilized metals being also partially removed, and the remaining button is treated with borax in O. F., the resulting glass is coloured usually with oxide of copper. This cold glass treated in R. F. on a fresh coal, becomes red and quite opaque on cooling, but sometimes, if the R. F. is kept up too long, the copper is reduced and leaves the glass colourless. The reaction succeeds better when the glass is treated a few seconds beside some tin in R. F.; part of the tin oxidizes at the expense of the oxide of copper, and dissolves without colouring the glass, while the resulting suboxide of copper makes the glass red and opaque. The lightness of the red colour depends upon the freedom of the glass from other colouring oxides. S. Ph. may be employed in place of borax.

When there is only a trace of copper, as in silver lead obtained on the large scale, or in lead reduced from cupreous

When the alloy contains much nickel, cobalt, iron, and arsenic, most of the Co and Fe can be separated by borax on coal in R. F., and recognized by the colour of the glass, after which lead may be added, and this, with the remainder of the cobalt and iron, dissolved in boracic acid, while the greater part of the arsenic will volatilize. The remaining cupriferous nickel button, which may contain some arsenic, is treated in O. F. with S. Ph., which will be dark-green while hot, becoming lighter on cooling, and when quite cold fine green, if not containing too little copper. The final green results from the yellow of the nickel and the blue of the copper.

To detect a little copper in tin it is treated with successive portions of S. Ph. on coal in O. F., until nearly all the tin is separated, and the remaining button imparts a bluish-green colour to the glass, when a bit of pure tin is added, and the glass treated a short time in R. F.; on cooling the bead becomes red.

Metallic sulphides.

Compounds of copper with sulphur and with metallic sulphides are roasted at a moderate heat on coal, with the O. F. and R. F. alternately, until all the sulphur is removed, and the product is then treated with soda in R. F., yielding metallic copper, or dissolved in the glass fluxes and tested for copper with tin on coal.

If other easily reducible metallic oxides are present, the reduction with soda affords an alloy of copper and other metals, which, if not in one button, may be obtained by washing away the slag and coal in the mortar, and is then refined with lead and boracic acid on coal, provided it contains no lead. When it is desirable to avoid refining, the alloy may be simply tested for copper with borax and S. Ph. as above described under metallic compounds. In case only sesquioxide of iron is present, the reduction does not afford an alloy, but the copper and iron are obtained apart, and can be clearly distinguished after washing, by the aid of the magnifier and the magnet. The presence of binoxide of tin, as in roasted stannite, causes a white, brittle alloy, which, treated for some time beside an S. Ph. bead in O. F. on coal, renders the bead red and opaque on cooling.

Should the roasted substance contain other colouring oxides besides copper, excepting those of bismuth and antimony, it will always yield the copper reaction as well as the other reactions, if dissolved in the fluxes in O. F. and then treated with tin in R. F., unless there is too little copper. In presence of much bismuth or antimony, however, the bead treated with tin becomes dark-gray to black on cooling and the red copper-colour is entirely concealed. When there is but little bismuth or antimony, the bead frequently becomes only brownish-gray. If a gray or black bead is obtained, the roasted substance must be fused in R. F. on coal, with a mixture of soda, borax, and test lead. The resulting button is then treated alone on coal, to volatilize the antimony, and afterward with boracic acid, until either a pure copper button remains, or all is dissolved, and the copper has imparted a blue, green, or red colour to the boracic acid; or else the copper button, after being freed from most of the lead and bismuth by means of the boracic acid, is tested with S. Ph. and tin as above.

When a substance consisting, for instance, chiefly of sulphide of iron, contains so little copper that it yields no reaction with borax or S. Ph. and tin, a larger quantity, about one hundred milligr., must be roasted as in the quantitative copper assay, then mixed with an equal amount of soda and half as much borax, and, in the absence of easily reducible metallic oxides, with thirty to fifty milligr. test lead, after which the mixture is reduced as in the quantitative assay. All the copper is contained in the resulting button, which is separated from the slag, and can be further treated for copper with boracic acid, and then with S. Ph. and tin. In place of the lead a bit of pure gold may be employed, which is afterward tested for copper with S. Ph.

The oxides of copper can be very easily recognized by testing with the glass fluxes, and by reduction with soda or neutral oxalate of potassa. When other metallic oxides or acids are also present, as in various metallurgical products, the remarks under iron are to be borne in mind. Antimony may be recognized partly by its coat on coal, and partly by the black bead afforded with S. Ph. and tin.

Silicates and other salts.

The silicates and other salts of copper dissolve in O. F. in the glass fluxes to green beads, blue on cooling, if free from other colouring oxides. The greater part of the silica remains undissolved in S. Ph. Treated with tin on coal the beads become red and opaque on cooling.

To obtain metallic copper, sulphates and arsenates must first be thoroughly roasted on coal, and the product, as well as the remaining salts, then reduced with soda and borax, when the copper generally unites to a single button, while the difficultly reducible oxides are dissolved in the borax. Phosphate of copper, however, only yields the whole of its copper when a bit of very fine iron wire is added to reduce the phosphoric acid.

When a compound of sulphates or arsenates of copper, nickel, cobalt, and sesquioxide of iron is roasted, the sulphur is volatilized, but part of the arsenic remains as arsenate of nickel, and when the roasted substance is reduced with soda and borax, copper, nickel, and arsenic unite to a fusible button, while the oxides of cobalt and iron dissolve in the borax. If the reduced button contains copper, it will at once impart to S. Ph. in O. F. a green colour, becoming somewhat lighter on cooling, and due to nickel and copper. The copper may further be tested for with tin.

The suboxide of copper in slags cannot be easily detected by means of soda and S. Ph., except in copper refining slags, because it forms frequently a very trifling ingredient, and the main constituents, which are silicates of the earths and difficultly reducible metallic oxides, conceal the copper reaction. It is therefore always necessary to reduce the slags with soda on coal. Should no copper be thus obtained, a larger quantity, one hundred milligr., must be treated with soda, etc., as directed for the roasted sulphide. Should everything be dissolved in the boracic acid, a very small quantity of copper may only produce a red, blue, or green colour at the spot where the last of the lead was dissolved; but if there is one per cent. of copper in the slag and the boracic acid is treated with the R. F. beside the lead, only the latter will be dissolved and the copper will remain, showing while melted its peculiar bluish-green colour. If the O. F. is then directed upon the copper, it will oxidize and colour the whole bead red, from suboxide. When the remaining button does not seem to be pure copper, it is fused beside S. Ph. in O. F. and the coloured bead treated with tin, as above.

In the forceps cupriferous minerals impart a green tinge to the flame, which is azure-blue in presence of chlorine, but afterward becomes green. When much lead is also present, the flame is blue with a green tip. Should copper not be thus simply detected, it may be found by moistening the substance first with hydrochloric acid, when the resulting chloride of copper affords an azure-blue, or greenish, and in certain cases reddish-blue colour. Silicates, *e. g.* slags, must be finely powdered, moistened with hydrochloric acid in a porcelain dish, dried over the flame, the powder stirred to a thin paste with water and then fused B. B. on platinum wire, when the azure-blue colour will appear, if copper is present.

b. Blowpipe characteristics of cupriferous minerals.

Metallic copper fuses to a bright, bluish-green globule, which is covered with black oxide on cooling. In O. F. shows oxide of copper reactions with the fluxes.

ARSENIDE OF COPPER.

Whitneyite, algodonite, and *domeykite* yield no sublimate in the closed tube. On coal they fuse readily to a bright globule, evolve arsenical fumes, and then react for copper with the fluxes.

Condurrite at first yields water in the matrass, then arsenous acid, and assumes on the surface a silver-white colour, inclining to bluish. In the open tube yields arsenous acid. On coal fuses easily, evolves a strong arsenic odour and yields a yellowish metallic mass, which reacts weakly for iron with borax in R. F. and then shows copper.

SELENIDES.

Berzelianite fuses readily to a gray, somewhat sectile button, and evolves an odour of selenium. In the open tube a red, pulverulent sublimate of selenium, bordered by a crystalline, easily volatile sublimate of selenous acid. Roasted and reduced with soda it yields copper. (Berzelius.)

Selenkupferquecksilber yields in the matrass mercury and selenium, and at a strong heat sulphurous acid can be detected. On coal volatilizes, affording a strong coat and odour of selenium, and leaves a trifling residue. By reduction affords traces of copper and iron.

Crookesite fuses very readily to a greenish-black, shining enamel, and colours the flame strongly green. (Dana.)

COPPER COMBINED WITH SULPHUR AND WITH OTHER SULPHIDES.

Chalcocite yields nothing to the closed tube; in the open tube sulphurous acid. On coal fuses readily to a globule, which spirts and evolves sulphurous acid. The powder reduced with neutral oxalate of potassa yields copper and sulphide of potassium, which sinks into the coal and is strongly hepatic.

Digenite yields traces of water and a sulphur sublimate in the closed tube. On coal yields some sulphur, otherwise like chalcocite.

Covellite yields traces of water and much sulphur in the closed tube. In the open tube sulphurous acid and, if quickly heated, sulphur. On coal burns with a blue flame, then behaves like chalcocite.

Bornite only becomes darker in the closed tube; in the open tube yields sulphurous acid. On coal fuses easily to a brittle, magnetic globule, with grayish-red fracture. The roasted powder yields iron and copper reactions.

Tennantite sometimes decrepitates slightly in the closed tube and yields sulphide of arsenic. In the open tube yields sulphurous and arsenous acids. On coal fuses easily, with intumescence and evolution of sulphur and arsenic fumes, to a dark-gray, magnetic globule. The roasted powder reacts for iron and copper, and by reduction yields copper buttons and metallic iron. No coat is perceptible on the coal.

Kupferblende, from Junge hohe Birke and Alte Mordgrube, near Freiberg, decrepitates very strongly in the closed tube and yields a little sulphur and, at a higher heat, sulphide of arsenic. On coal like tennantite, but is distinguished by affording a coat of arsenous acid and, with R. F., an abundant zinc coat. Roasted in powder it reacts for copper and iron, and by a reduction assay affords a strong zinc coat, while copper and iron remain after the coaly matters are washed away. Upon separating the iron with the magnet and cupelling the copper with lead, a little silver is obtained.

Enargite decrepitates with some violence in the closed tube and yields a sulphur sublimate at a gentle heat; more strongly heated it fuses to a button, and sulphide of arsenic is given off. In the open tube the powder yields sulphurous and arsenous acids, the latter mingled with oxide of antimony. On coal the powder fuses readily to a globule and affords slight coats of arsenous acid and oxides of antimony and zinc.

On pulverizing and roasting this globule, dissolving it in borax and reducing out the copper, a little iron may be detected by the greenish colour of the borax glass in R. F. and the yellow colour assumed by it when fused again in O. F. on platinum wire.

Barnhardtite and *homichlin* yield a sulphur sublimate in the closed tube; otherwise like bornite.

Tetrahedrite sometimes decrepitates in the closed tube, fuses, and strongly heated B. B. yields a dark-red sublimate of sulphide with oxide of antimony, or sulphide of arsenic, or a mixture of both, according as $\overset{'''}{Sb}$, $\overset{'''}{As}$, or both together are present. A low red-heat suffices to produce a dark-gray to black sublimate of Hg, if present.

In the open tube fuses and yields copious antimonous fumes and sulphurous acid, frequently also arsenous acid: the residue is black and infusible. If $\acute{H}g$ is present, a mirror of mercury forms before any considerable antimonous fumes appear; by too rapid heating black $\acute{H}g$ is sublimed.

On coal fuses readily to a globule and yields a copious antimony coat, while with a good R. F. a second coat is formed, yellowish while hot, white on cooling, and showing zinc with cobalt solution, p. 230. In presence of lead this cannot always be detected. Arsenic is recognized by the odour, unless in too small quantity, when some soda must be mingled with the powdered mineral and the whole treated in R. F. The sulphur is thus kept back and the arsenic alone volatilized. The globule remaining after treating the mineral alone on coal is pulverized, roasted, and tested with glass fluxes and with soda, when it yields reactions for iron and copper. Reduction with soda and borax sometimes affords niccoliferous copper.

To detect a very little mercury, it may be necessary to heat the fine powder, mixed with three volumes of dry soda, or neutral oxalate of potassa, in a matrass. Silver is found by an assay with test lead (*vide* quantitative silver assay).

Binnite yields in the closed tube sulphide of arsenic, in the open tube sulphurous and arsenous acids. On coal yields an arsenic coat and odour, and fuses with spirting to a black globule, surrounded by a zinc coat. With the fluxes gives copper reactions.

Annivite behaves like plumbiferous tetrahedrite: bismuth can only be found by the wet way.

Fieldite, probably like enargite with antimony predominating.

Aftonite like argentiferous tetrahedrite.

Fournetite like plumbiferous tetrahedrite.

Chalcopyrite decrepitates in the closed tube, yields sulphur and becomes dark or tarnished; in the open tube evolves sulphurous acid copiously. Fuses on coal, spirting and throwing off sparks, to a black, rough, magnetic globule, with a dark-gray fracture. Well roasted gives iron and copper reactions; by reduction with soda yields iron and copper.

Chalcostibite decrepitates in the closed tube, fuses, and at a high heat yields a little sulphide of antimony. In the open tube evolves sulphurous acid and copious antimonial fumes. On coal fuses readily, with evolution of antimonial fumes, to a globule, which tested with borax shows iron. The remaining globule yields copper with soda.

Cubanite yields traces of sulphur in the closed tube; in the open tube sulphurous acid. Fuses readily on coal, with evolution of sulphurous fumes, to a magnetic globule. Lead, if present, produces a feeble coat. Roasted in powder it yields iron and copper reactions with the fluxes, and metallic iron and copper by a reduction assay.

Wölchite yields some water in the matrass and fuses with evolution of sulphur and sulphide of arsenic, to a reddish-brown scoria. On coal fuses with ebullition and deposits antimony and lead coats, while a lead-gray metallic button remains, which after roasting yields with soda a copper button.

COPPER COMBINED WITH CHLORINE.

Atacamite in the closed tube yields water rather abundantly and gives a gray sublimate, becoming grayish-white on cooling. Fuses on coal, affording an azure-blue flame with a green tip, and gives two coats, one brownish and one grayish-white. It is reduced to a copper button, surrounded by some slag. Under R. F. the coats change their position, and show the azure-blue flame of chloride of copper.

Percylite decrepitates in the closed tube, and when gently heated assumes a transient green colour; little water is obtained, and the mineral finally fuses to a brown fluid. B. B. in the forceps in O. F. gives a green flame with a dark-blue tip; on coal in R. F. alone, or with soda, affords copper and lead buttons.

OXIDES OF COPPER.

Cuprite in the forceps fuses and colours the flame emerald-green; moistened with hydrochloric acid yields an azure-blue flame. On coal blackens, fuses, and is reduced to copper, which is coated with a thin film of black oxide on cooling.

Melaconite, like oxide of copper (see tables).

Kupferschwärze sometimes yields much water in the matrass. Alone on coal is reduced to a copper button, frequently surrounded by slag. With the fluxes affords reactions for copper, iron, and manganese.

OXIDE OF COPPER COMBINED WITH ACIDS.

The *sulphates of copper* behave as follows:—

Brochantite yields water and sometimes blackens in the matrass. If then powdered, mixed with charcoal dust and strongly heated in the closed tube, it evolves sulphurous acid. On coal is reduced with effervescence to a copper button.

Langite.—B. B. on coal yields water, acid fumes and metallic copper. Heated it becomes bright green, losing one equivalent of water, then various tints of olive-green, and lastly black. It has, finally, a strong acid reaction. (Dana.)

Chalcanthite swells in the matrass, yields water and whitens. When mixed with coal dust and heated in the closed tube, it evolves sulphurous acid copiously.

On coal colours the flame green, fuses and is reduced with effervescence to a copper button, coated with sulphide of copper. When well roasted, reacts for copper and sometimes iron with the fluxes. With soda yields copper.

Among the *phosphates*, *pseudomalachite*, *dihydrite*, *ehlite*, and *libethenite* behave as follows:

In the matrass they yield water, blacken, and sometimes decrepitate if quickly heated. After ignition in the matrass a fragment heated in the forceps fuses without colouring the flame perceptibly; the fused globule is black and has a crystalline surface. Gradually heated on coal (in powder if they decrepitate) they blacken and fuse to a button, with a core of metallic copper. With the glass fluxes they react like oxide of copper. A sufficient quantity of soda causes their reduction to metal in a strong flame, but with a little soda they swell up and fuse to a globule. This phenomenon is repeated on each fresh addition of soda, until finally a swollen mass results, which only fuses in a strong flame, spreads out, sinks mostly into the coal, and leaves metallic copper. Arsenic acid may be detected in some of them by fusion with soda.

Berzelius has proposed a test for phosphoric acid in these minerals, founded upon the peculiar behaviour of phosphate of copper with metallic lead. When to the phosphate fused on coal an equal volume of lead is added and the whole melted together for some time in a good flame, the copper separates as metal, around which is a fluid mass of phosphate of lead, which is crystalline when cold. If the metallic button is separated from this new compound and the latter treated alone in R. F., a perfectly round bead finally results, which on cooling crystallizes with large facets and usually has a red colour (Ꞓu). This behaviour shows that phosphoric acid has a greater affinity for oxide of lead than for oxide of copper.

Tagilite probably like the preceding.

Thrombolite yields much water and blackens in the matrass. In the forceps fuses easily, colouring the flame at first azure-blue, probably from chloride of copper, but afterward emerald-green. On coal fuses easily to a black globule, which finally spreads out and shows metallic copper buttons. With glass fluxes gives only copper reactions; with boracic acid and iron affords phosphide of iron.

The *carbonates of copper* behave as follows:

Malachite and *azurite* yield water and blacken in the matrass. Fuse to a button on coal and are reduced to metallic copper. With the fluxes behave like oxide of copper, and dissolve with effervescence in hydrochloric acid.

The *arsenates* behave as follows:

Clinoclasite, from Cornwall, yields a little water in the matrass, and behaves like olivenite.

Olivenite yields some water in the matrass. In the forceps fuses to a globule and colours the flame bluish-green. On cooling it has a crystalline appearance. On coal it fuses, with deflagration and evolution of arsenical fumes, to a somewhat brittle, brown-metallic globule, with a white fracture, which treated with lead yields the phosphoric acid reaction just described. The detached button of lead and copper leaves pure copper when treated with boracic acid (see copper assay).

Cornwallite yields water in the matrass. On coal evolves arsenical fumes, and fuses to a globule of copper surrounded by a brittle crust.

Erinite frequently decrepitates very strongly and yields much water in the matrass. On coal in powder is reduced, with evolution of arsenical fumes, to a brittle globule with a grayish fracture, which in O. F. affords pure copper. In presence of phosphoric acid a trifling crystalline slag remains with the copper.

Euchroite yields considerable water, and assumes a darker green in the matrass; otherwise like olivenite.

Chalcophyllite decrepitates very strongly in the matrass, yields much water and breaks into fine olive-coloured scales. On coal like olivenite.

Tyrolite decrepitates, yields considerable water and blackens in the matrass; the assay afterward fuses to a steel-gray bead in the forceps. On coal evolves arsenical fumes and fuses to a gray scoria, in which copper globules are formed by R. F. After reducing out all the copper with borax and soda, and then dissolving the slag in hydrochloric acid, considerable lime may be detected by adding oxalic acid to the solution, after it is made ammoniacal. The lime is present as carbonate, as is shown by the effervescence of the mineral with warm nitric acid.

Trichalcite behaves like cornwallite.

Liroconite yields much water and becomes dark olive-green in the matrass. In the forceps fuses and colours the flame bluish-green. On coal fuses, with formation of bubbles and evolution of arsenical fumes, to a dark-brown slag, which contains copper globules and with borax and soda affords an arsenical copper button. In the slag alumina, partly combined with P, can be detected.

Conichalcite decrepitates strongly, yields water and blackens in the matrass. On coal deflagrates, evolves a slight arsenic odour, and forms a red slag, which has an alkaline reaction on litmus-paper. In the forceps fuses, colouring the flame at first strongly green, but afterward only green at the extremity, while near the assay it has a feeble light-blue colour. With borax in O. F. a yellowish-green bead, blue on cooling. With S. Ph. and lead on coal in R. F. affords a glass, dark-yellow while hot and chrome-green on cooling (vanadic acid). With soda on coal in R. F. effervesces, evolves arsenical fumes and fuses to a globule; on longer blowing affords a copper button and a white earthy mass. Lime and phosphoric acid may be detected by the wet way.

Vanadates of copper, volborthite, and *Kalkvolborthite* yield water and blacken in the matrass. On coal yield a black slag containing copper buttons, and if reduced with soda afford copper. With the fluxes give copper reactions. The vanadic acid is found according to vanadium examination.

Cuproscheelite blackens in the closed tube, and gives off water. B. B. fuses on the edges to a black glass, and colours the flame an intense green. On charcoal blackens, fuses with a little intumescence, forming finally a slag containing minute particles of metallic copper. With fluxes gives copper and tungstic acid reactions. (Dana.)

The silicates, *dioptase* and *chrysocolla,* yield water and blacken in the matrass. In the forceps are infusible, but colour the flame intensely green. With the fluxes yield copper reactions, and with S. Ph. a silica skeleton. On coal they blacken in O. F., become red in R. F., and with soda in R. F. yield copper buttons.

c. *Metallurgical products.*

The method of examining metallurgical products for copper may be inferred from the general remarks on examination for copper.

13. MERCURY, Hg.

Its occurrence in the mineral kingdom and in metallurgical products.

Mercury occurs under the following circumstances in nature:

a. Metallic;
b. With *selenium;*
c. Combined with *sulphur;*
d. Combined with *chlorine;*
e. Combined with *iodine* in coccinite from Mexico;
f. With *antimonic acid* and *sulphur.*

Mercury also forms an ingredient of certain amalgamation products and residues, including—*gold* and *silver amalgam*; unwashed *amalgamation residues*, which frequently contain finely-divided amalgam and *subchloride of mercury*. When the ores contain lead or copper, the residues may often contain a little *lead* and *copper amalgam*.

Examination for Mercury.

a. General examination for mercury.

Compounds of mercury with gold and silver, including *native* and *artificial amalgams*, and also *residues* not yet purified from silver, copper, and lead amalgam by washing, are heated over the spirit-lamp in a matrass formed by blowing a bulb on the end of a glass tube (see figure). Generally a bit of amalgam as large as a millet-seed will suffice, but in case of the residues from amalgamation the bulb should be at least half filled. Any water mechanically combined will be expelled by the first action of the heat, and should be wiped away with blotting-paper. On heating to redness, the mercury separates in vapour, which condenses on the colder part of the tube at *a* to small metallic globule, that cannot be mistaken for any other metal. The metals and residue in the matrass may then be further tested for gold, silver, etc.

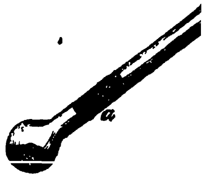

Fig 65

Selenide of mercury may be recognized by the lustrous, crystalline, gray sublimate formed in the neck of the matrass. If mixed with much soda, the mercury separates in globules, leaving the selenium combined with sodium.

Combined with sulphur, as in *cinnabar*, mercury affords a black sublimate, which assumes a red colour by friction. Powdered cinnabar heated with three volumes of soda, previously dried by bringing it to incipient redness in the platinum spoon, yields a sublimate of metallic mercury and a little cinnabar, while the sulphur remains combined with sodium. If neutral oxalate of potassa, or, better still, a mixture of the oxalate with cyanide of potassium, is employed, metallic mercury alone is obtained.

Artificial cinnabar, or vermilion, if adulterated with minium, leaves in the matrass a residue of sulphide of lead, which can be recognized on coal. The same remarks apply to an admixture of sulphide of antimony.

When sulphide of mercury is combined with other sulphides, as in certain varieties of tetrahedrite, even when very little is present, the test in the matrass affords, by the first action of the heat, a black sublimate of this sulphide, because it is so volatile that it can readily be separated from the other sulphides by an elevated temperature. Upon mixing the powdered substance with neutral oxalate of potassa and cyanide of potassium, and heating it to redness in a narrow-necked matrass, metallic mercury separates and condenses on the neck to a gray coat, which may be united to a globule by gently tapping on the glass, unless too little mercury is sublimed. Should there be so little mercury that no film can be detected with certainty, the assay may be repeated and the end of an iron wire wrapped about with a bit of pure gold leaf held near the mixture while heating it. The gold will become entirely white, or at least very perceptibly so, if the slightest trace of mercury is present.

Chloride of mercury.

Combined with chlorine, mercury affords a white sublimate in the matrass, or if mixed with dry soda or neutral oxalate of potassa, metallic mercury volatilizes on heating, leaving a chloride of the alkali. To detect small quantities of the chlorides of mercury in substances, it is only necessary to heat them with soda or oxalate of potassa, observing the directions given for detecting a trifling amount of sulphide of mercury.

Iodide of mercury (Hg I) fuses very easily in the matrass and affords a crystalline, yellow sublimate, red on cooling. *Subiodide of mercury* (Hg^2 I) fuses and sublimes unaltered, when quickly heated; if slowly heated, it is decomposed into mercury and the iodide. In the matrass with soda or neutral oxalate of potassa both compounds yield mercury.

Salts.

The oxy-salts of mercury are also best decomposed by ignition in the matrass with perfectly dry soda, or neutral oxalate of potassa, which causes the separation of metallic mercury.

b. Blowpipe characteristics of the mercuriferous minerals above named.

Native mercury sublimes in the matrass and condenses to small globules, which can be readily caused to unite; if too strongly heated, it boils and spirts. Any silver present will remain behind, and can then be cupelled with lead (*vide* silver).

Amalgam and *arquerite* gradually heated to redness in the matrass, afford mercury and leave spongy silver, which may be fused to a button on coal, or if impure may be cupelled.

MERCURY COMBINED WITH SELENIUM.

Tiemannite decrepitates in the closed tube, swells and fuses, volatilizing completely when pure and forming a black sublimate, brownish-red at the side farthest from the assay. The residue from impure fragments reacts for iron and silica with fluxes. The addition of considerable soda causes mercury to

In the open tube it affords a selenium odour and a black sublimate, followed by a reddish-brown one and then a white sublimate of selenite of mercury, sometimes fusible to drops like tellurous acid. On coal it volatilizes with an azure-blue flame, and affords a lustrous metallic coat, surrounded by a dark-brown coat. (Kerl.)

Onofrite (*Selenschwefelquecksilber*), according to H. Rose, volatilizes unchanged, yielding a black sublimate of mixed sulphide and selenide of mercury. With soda it affords mercury and on coal diffuses a selenium odour.

Onofrit, a yellow, earthy mineral, volatilizes with a selenium odour and affords a sublimate of mercury and some yellow compound. (Köhler.)

SULPHIDE OF MERCURY.

Cinnabar yields in the matrass a dark sublimate, which gives a red streak. If there is any residue, it occasionally reacts for iron, copper, and lead.

In the open tube it affords sulphurous acid and mercury, but if quickly heated, part of it sublimes unchanged.

On coal volatilizes completely if pure.

Hepatic cinnabar affords in the matrass a very dark sublimate of cinnabar, evolves a distinct sulphuretted hydrogen odour and leaves a black coaly mass, which on ignition in the open tube, or on platinum foil, gradually disappears, leaving only a trace of earthy matter.

Calomel yields the reactions of mercury and chlorine, as given on p. 275. On coal volatilizes and forms a white coat, and with S. Ph. and oxide of copper shows chlorine (*vide* chlorine).

Iodide of mercury (artificial) yields violet iodine vapours when heated with bisulphate of potassa in the matrass. (See also under the general examination for mercury.)

c. *Metallurgical products.*

The essential points regarding the examination for mercury n these products are given under the general examination.

14. SILVER, Ag.

Its occurrence in the mineral kingdom and in metallurgical products.

Silver occurs in nature :—

a. Metallic and *alone ;*

b. Combined with other metals;

c. Combined with *selenium* and other *selenides ;*

d. With *sulphur* and other *sulphides ;*

Silver combined with sulphur also occurs in trifling quantity in many lead and copper ores, as galena, several of the plumbiferous minerals and copper ores. Pyrite, arsenopyrite, and zinc-blende not unfrequently contain a little sulphide of silver.

e. Combined with *chlorine* and *bromine ;*

f. With *bromine ;*

g. With *iodine.*

Silver occurs in metallurgical products as follows :

a. Metallic in

Refined silver, frequently with traces of Pb, and sometimes Au and Cu;

Brightened silver, containing a little Pb, Cu, and sometimes Bi, Sb, As, and Ni;

Cement silver from the "extraction" of argentiferous ores and products; it frequently contains more or less Pb and trifling quantities of other metals;

Retort silver, often containing more or less Cu, traces of Au, Ni, Co, and, before refining, also Fe, Zn, Sb, Pb, As, and Hg;

Amalgam, usually containing the ingredients of the foregoing product;

Raw, or silver lead, almost always containing trifling quantities of other metals (*vide* lead).

A little Ag also occurs in black copper, raw copper, and refined copper (*vide* iron); as well as in many specimens of lead (*vide* lead).

b. Combined with *sulphur* it occurs in trifling quantity in the various *matts* and *speiss-like products* from smelting silver, lead, and copper ores, and in certain furnace deposits (*vide* iron).

c. As *oxide* in very small quantities in the products of cupellation, viz., *litharge*, *abzug*, *abstrich*, and *cupel bottoms* or *hearth* (*vide* lead). Here should also be mentioned the *test mass*, or bottom of the silver-refining hearth.

Slags containing silver often owe its presence chiefly to fine, disseminated particles of argentiferous matt, although some slags contain silicate of silver.

Examination for Silver.

a. General examination for silver.

Compounds of silver with metals, volatile at a high heat, as *antimony*, *lead*, and *bismuth*, yield a coat on coal. After nearly the whole of these metals have been volatilized by long blowing, the coat becomes reddish to carmine-red from oxide of silver, if there is not too little of that metal present, and the remaining button shows a more or less pure silver colour. This reddening of the coat is highly characteristic, and may always be regarded as indicating silver. In combination with much lead or bismuth, silver is found by

Alloys.

cupellation, with addition of test lead, if that is made necessary by the presence of other oxidizable metals (*vide* quantitative silver assay).

When silver containing *arsenic* is treated alone, on coal, the arsenic volatilizes and may be recognized by its odour.

Selenium behaves similarly. *Tellurium*, if present in considerable quantity, partly volatilizes and coats the coal, but part of it remains persistently with the silver, and can only be removed by treating the compound, pulverized as much as possible in the R. F., with soda, or neutral oxalate of potassa. *Mercury* combined with silver can be removed by ignition on coal, or in the matrass; on coal the silver fuses to a button, in the matrass it forms a porous residue.

If the silver is combined with much *gold* and the alloy is fused in O. F. with S. Ph. on coal, the silver oxidizes and is gradually dissolved, rendering the glass opalescent when cold. By separating this glass from the gold button and treating it alone on coal in R. F., the oxide is readily reduced and united to a button of silver.

When non-volatile metals, more easily oxidized than silver, are present, viz., *copper*, *nickel*, and *cobalt* in not too small quantity, they may readily be detected by testing with borax or S. Ph. on coal, and frequently so far separated as to leave the silver with a pure surface. When present in large quantities, they can only be entirely separated by cupelling the silver with test lead. Should these metals be present in so small proportions that no distinct reactions are obtained with the glass fluxes, a sufficient quantity of the silver is treated first on coal alone in O. F. and notice taken of any coat that may be formed. After this the button is dissolved in nitric acid in a test tube, diluted with water, a few drops of hydrochloric acid added, and the whole well shaken, so that the chloride of silver may settle. When the fluid is clear, a drop or two of hydrochloric acid is added, to ascertain whether it will produce any further cloudiness. If it does, the whole must again be well shaken; if not, the solution may be filtered at once, the filtrate heated to boiling in a porcelain vessel and a solution of potassa gradually added to feebly alkaline reaction. By this means the other metals, present as oxides, are thrown down, and after filtration may be readily detected by means of the glass fluxes.

The chloride of silver can be reduced on coal with soda.

Sulphides. Minerals and metallurgical products consisting of, or containing sulphides of metals, and which are to be directly examined for silver, may be most advantageously treated according to the method given under the quantitative silver assay, and the silver lead cupelled. If rich in silver, but little of the substance need be treated, while, if poor, as much should be used as for the quantitative assay. The quantity of test lead and borax glass depends partly upon the quantity of the substance taken and partly upon its character; whether it contains difficultly fusible ingredients which are to be slagged off, or contains copper, nickel, etc. Most of the above-mentioned minerals containing sulphides show the presence of silver by the reddish coat formed on coal.

b. Blowpipe characteristics of argentiferous minerals.

Native silver fuses on coal to a bright globule, which has a silver-white colour on cooling. The presence of antimony causes a feeble white coat of antimonous acid, which afterward becomes red (p. 65). Arsenic, if present, is detected by the odour during fusion. With borax in R. F. on coal it sometimes affords a glass that reacts for cobalt and iron.

Chilenite fuses easily, evolves a slight arsenical odour, and coats the coal with oxide of bismuth. By cupellation affords a silver button, and stains the bone-ash dark-green with oxide of copper.

Dyscrasite fuses very readily to a button on coal, affords a copious coat of oxide of antimony, which afterward reddens, and finally a rather pure silver button remains.

Hessite fuses in the open tube, without affording very copious fumes. On coal fuses readily to a globule, and yields part of its tellurium, coating the coal; but the greater portion remains with the silver. When cold the surface is covered with lustrous metallic globules.

Fused in a fine state with soda, or neutral oxalate of potassa in R. F., the tellurium is separated, leaving the silver in little globules, which if cleansed from the coal and slag by washing, and then dissolved in nitric acid, generally leaves a little gold behind.

SILVER IN COMBINATION WITH SELENIUM.

Naumannite fuses and yields a trifling sublimate in the closed tube. On coal in O. F. fuses quietly, but in R. F. intumesces, and in solidifying glows again. With soda and borax affords a lustrous silver button. (G. Rose.)

Eucairite.—In open tube like berzelianite (p. 266). On coal fuses with a strong odour of selenium, and affords a gray, soft, but not malleable, metallic button. Cupelled with test lead, leaves a silver button, and with the fluxes reacts strongly for copper. (Berzelius.)

SILVER IN COMBINATION WITH SULPHUR.

Argentite and *acanthite* fuse on coal in O. F., with intumescence and evolution of sulphurous acid, affording at length a silver button. If impure, a slag also results, which reacts usually for iron, and sometimes for copper. With soda a silver button is very easily obtained.

Stephanite decrepitates in the closed tube, then fuses, and after some time yields a slight sublimate of sulphide of antimony. In the open tube fuses and evolves antimonous fumes and sulphurous acid. On coal fuses very easily, with spirting, coats the coal with oxide of antimony, and is converted into sulphide of silver, containing but little antimony. On long blowing, the coat reddens and a silver button remains, sometimes with a scoria that reacts for copper and iron.

Polybasite fuses with extraordinary ease in the closed tube, but yields no sublimate. In the open tube fuses and yields sulphurous and antimonous fumes. Under the magnifying glass, if $\overset{\prime\prime\prime}{As}$ was present, the sublimate is seen to consist of antimonous acid mixed with crystalline arsenous acid. On coal in O. F. fuses very readily, with spirting to a globule, which evolves sulphurous acid and coats the coal with oxide of antimony, and, in presence of arsenic, arsenous acid.

Long blowing occasionally produces a yellowish-white zinc coat near the assay, and finally a metallic, mirror-like button is obtained. The cold button has a black surface, and the white coat is somewhat reddened by oxide of silver. With S. Ph. the button behaves like cupriferous silver.

Proustite fuses very readily in the closed tube, and at incipient redness affords a slight sublimate of sulphide of arsenic. The residue has a dark, lead-gray, scaly fracture and feebly metallic lustre. In the open tube affords sulphurous and arsenous acids, with some antimonous fumes, if $\overset{\prime\prime\prime}{\mathrm{Sb}}$ replaces some $\overset{\prime\prime\prime}{\mathrm{As}}$. Fuses on coal with evolution of sulphurous and arsenous fumes, and coats the coal with arsenous acid (and, in presence of $\overset{\prime\prime\prime}{\mathrm{Sb}}$, with oxide of antimony); but later the fused globule behaves like sulphide of silver. This fused for some time in O. F., or reduced with soda, affords pure silver.

Xanthoconite in the closed tube assumes a transient, dark-red colour. More strongly heated fuses, and then behaves like proustite. In the open tube and on coal like proustite free from antimony.

Pyrargyrite fuses very readily in the closed tube, sometimes flying into small pieces at first, and at a continued red heat affords a sublimate of amorphous tersulphide of antimony. Fuses in the open tube, and evolves sulphurous acid and antimonial fumes. On coal fuses very readily with spirting to a globule, yields sulphide of antimony, coats the coal with oxide of antimony, and is converted into sulphide of silver, which obstinately retains some antimony. By long treatment in O. F., or reduction with soda, this affords pure silver. Any $\overset{\prime\prime\prime}{\mathrm{As}}$ replacing $\overset{\prime\prime\prime}{\mathrm{Sb}}$ can be recognized by the odour when the powdered mineral, mixed with soda or neutral oxalate of potassa, is fused on coal in R. F.

Pyrostilpnite; in the open tube and on coal like pyrargyrite.

Stromeyerite fuses very easily in the closed tube, and only rarely yields a little sulphur. In the open tube fuses to a globule and evolves sulphurous acid, but no sublimate when pure. Fuses very readily to a globule on coal, and in O. F. evolves only sulphurous acid if pure. The fused globule has a metallic lustre, is half malleable, and has a gray fracture.

With the fluxes reacts strongly for copper and sometimes feebly for iron. Cupelled with test lead, it leaves a silver button and a dark-green copper stain on the cupel.

Jalpaite, like the preceding.

Miargyrite decrepitates in the closed tube, fuses very easily and gives a film of sulphide of antimony. In the open tube evolves sulphurous acid and abundant antimonous fumes. On coal fuses very readily and quietly, with emission of sulphurous and antimonous fumes, to a gray globule, which finally in O. F. changes to a bright silver button, while the antimony coat reddens. Treated with S. Ph. and tin, the button shows a feeble but distinct reaction for copper.

Sternbergite yields in the open tube only sulphurous acid; on coal fuses with evolution of sulphurous acid to a globule, which is covered with metallic silver and is magnetic. Roasted in powder on coal and treated in R. F. with borax, gives a silver button, and a black opaque glass, which reacts for iron.

Freieslebenite in the open tube yields sulphurous acid and antimonous fumes, the non-volatile portions of which likewise contain antimonate of lead. On coal fuses easily, forming at a certain distance a coat of oxide of antimony mixed with sulphate of lead, and nearer the assay a dark-yellow coat of oxide with antimonate of lead. The coat finally becomes very red, and a silver button is left, which may be purified by treatment with boracic acid on coal.

Lichtes Weissgiltigerz from Freiberg.—Fuses in the open tube, and behaves like freieslebenite. On coal fuses very easily, spreads out, coats the coal strongly with oxides of antimony and lead, and leaves small, grayish-white, metallic buttons. The lead coat conceals the oxide of zinc. Fused with borax in R. F., the metallic buttons unite to a small silver button, showing copper with S. Ph., while the borax glass appears bottle-green from iron.

Schapbachite.—Fuses readily on coal, giving lead and bismuth coats, evolving sulphurous acid and leaving a silver button, which retains a little lead, bismuth, and copper.

COMPOUNDS OF SILVER WITH CHLORINE, BROMINE, AND IODINE.

Chloride of silver (*cerargyrite*), on coal in O. F. fuses very easily (frequently with ebullition) to a globule, which is pearl-gray, brownish, or black, according to the purity of the mineral. In R. F. gradually reduced to metal; immediately with soda. With oxide of copper on coal gives the chlorine reaction. Fused with bisulphate of potassa in a matrass, it unites beneath the fluid salt to a bead, which is white when cold. If the salt is dissolved by warming it with water, and the remaining bead of chloride of silver well dried between filter-paper and exposed to the sunlight, it soon assumes a gray or violet colour.

The compounds of silver with *chlorine* and *bromine* (*embolite*) fuse very easily on coal, spread out and are gradually reduced to metallic silver, evolving a suffocating odour. Soda greatly accelerates the reduction. If the alkaline mass is then broken out from the coal, dissolved in water, evaporated to dryness, and the dry salt fused in a matrass with bisulphate of potassa, the compounds rich in bromine emit reddish-yellow bromine vapours.

With oxide of copper on coal they tinge the flame greenish at first, but afterward intense blue.

Fused with bisulphate of potassa in the matrass, they unite beneath the fluid salt to dark red drops, which are light lemon-yellow when cold. Treated like the chloride mentioned just above, and exposed to the sunlight, the compounds in which chloride of silver prevails assume a greenish-gray colour, while those containing more bromide appear dirty green.

Bromide of silver (*bromyrite*) on charcoal emits pungent bromine vapours, and yields a button of silver. The artificial bromide fuses easily, and with oxide of copper in R. F. colours the flame greenish at first, but afterward intense greenish-blue.

In the matrass fuses very readily and has a quite dark red colour while fluid, becoming lighter on solidifying, so that when cold it has a deep yellow colour and is nearly transparent. Fused with bisulphate of potassa it evolves very trifling bromine vapours, and the fluid salt surrounding the bromide of silver assumes a yellow colour. Treated like the chloride of silver above, it gradually assumes in the sunlight a dark asparagus-green colour.

Iodide of silver (*iodyrite*) fuses in the closed tube and assumes a deep orange-colour, but resumes its yellow colour on cooling. B. B. on coal gives fumes of iodine and a globule of metallic silver. (Dana.)

The artificial iodide fuses on coal with extraordinary ease, spreads out, evolves a pungent iodine odour, tinges the flame reddish, and very soon leaves metallic silver globules. With oxide of copper in R. F. colours the flame green.

In the matrass alone acts like iodyrite. Fused with bisulphate of potassa, it unites to a dark red drop, below the salt, and evolves iodine vapours. Treated like the chloride and bromide above, its yellow colour remains unchanged.

c. Examination of metallurgical products.

The method of examining the products above enumerated for silver may be deduced from the remarks under the examination for silver in general.

15. PLATINUM, Pt; PALLADIUM, Pd; RHODIUM, Rh; IRIDIUM, Ir; RUTHENIUM, Ru; AND OSMIUM, Os.

Their occurrence in the mineral kingdom.

They are found:—

a. Metallic.

Two of the above-mentioned metals are also found as

b. Oxides in

Irite, probably $(\text{Ir}, \text{Os}, \text{Fe},) + \cancel{\text{Ir}}, \cancel{\text{Os}}, \cancel{\text{Cr}})$, with 55·2 Ir, 9·3 Os, 10·5 Fe, 10 Cr.

c. Combined with *sulphur* in

Laurite, perhaps—$12\ \text{Ru}^2\ \text{S}^3 + \text{Os}\ \text{S}^4$. (Dana.)

According to Pettenkofer, platinum is very widely distributed, and all the silver in commerce is said to contain it.

Blowpipe characteristics of the above-mentioned minerals.

Platinum and *platiniridium* are, B. B., infusible combinations of different metals which cannot be so decomposed, by further treatment B. B., that the presence of each metal may be proved by a definite reaction.

When tested with borax or S. Ph., they do not fuse, and are not oxidized or dissolved; in this operation, if the mineral has been filed off in a fine powder, more or less coloured beads, it is true, are obtained; but this colour comes from the admixture of oxidizable metals, viz., iron and copper, which may be found in this manner.

If the same compounds are fused on coal with borax and test lead, and a cupellation of the button commenced, the latter operation lasts only so long as the infusible metals permit. There results finally an infusible combination, in which there is still much lead, and which therefore possesses a lustrous surface and is somewhat brittle. If a medium-sized gold button is added, and the cupellation performed with a stronger heat, a bright metallic button can be sometimes obtained, perfectly free from lead, and of a yellowish-white or even platinum-gray colour. If it cannot be cupelled fine upon the cupel, it must be treated on coal with vitrified boracic acid, as follows: a shallow cavity is bored in the cross section of a good coal, or in a charcoal capsule, the button containing lead laid in it, covered with a little vitrified boracic acid, and the whole treated with the point of the blue flame. When the button is fused, the coal is inclined, so that the former comes out from under the melted glass, but still remains in contact with it, thus affording a large surface for oxidation. The point of the flame is now directed for a long time uninterruptedly upon the fluid glass, the metal being freely exposed to the air; in this way all the lead is oxidized and dissolved in the boracic acid, and the surface of the button becomes bright.

The resulting alloy of gold, platinum, rhodium, iridium, palladium, and osmium (the iron and copper having been removed), is hammered thin, ignited on coal, and dissolved in aqua regia, which leaves the finely divided, black, metallic iridium behind. The solution is poured into a porcelain dish, treated with as much chloride of ammonium as is necessary to alter all the platinum into platinum-chloride of ammonium, and the whole carefully evaporated to dryness at a gentle heat, in order to prevent any partial decomposition of the salts which have been formed. The dry salts are brought upon a filter and washed with alcohol of 60° to 70°, until no yellow colour is imparted to fresh alcohol. By this operation the gold, with other soluble salts, is dissolved out, and after water has been added to the solution and the alcohol driven off by heating, can be precipitated as metal from the warm solution by sulphate of iron. The residuary double salt is bright yellow, and is altered to spongy platinum by heating to redness in a platinum spoon. In such small assays no notice can be taken of the minute quantities of Rh, Pd, and Os, which are present in the native platinum.

The metallic precipitate of gold can be fused on coal, with the addition of a little borax or S. Ph., to a button, which is generally quite pure. If, however, it does not appear perfectly pure, it is fused on coal in R. F. with some borax and three parts of pure silver, the alloy treated first with nitric acid, and then the residuary gold, after washing, with bisulphate of potassa, as will be described in detail under the quantitative gold assay, for gold containing rhodium. In this way all the metals that might be present are entirely separated from the gold, and if this, when well boiled with water, is melted on coal to a button, it will appear perfectly pure.

If native platinum is dissolved in aqua regia, the blood-red solution evaporated almost to dryness, the acid fluid diluted with water and a few drops of a solution of potassa added, a yellow precipitate is formed, which consists chiefly of platin-chloride of potassium.

Palladium.—The behaviour B. B. of this native metal is not known.

Palladium which has been reduced from the oxide, but has not yet been hammered, behaves, according to Berzelius, as follows:

Carefully heated on platinum foil to low redness, it acquires upon the surface a blue colour, which, however, disappears at full redness. On coal alone, it is infusible and unchangeable. With sulphur in R. F., it fuses, but in O. F. the sulphur burns off, leaving the palladium behind. When fused with bisulphate of potassa in a sufficiently large matrass, it is dissolved with the evolution of sulphurous acid. The salt appears yellow when cool.

Iridosmine, light-coloured, is infusible B. B. Fused in a matrass with nitre, vapours of osmic acid are evolved, which can be very distinctly recognized by their unpleasant odour.

Iridosmine, dark-coloured, is infusible B. B., but gives an odour of osmium; it is, according to G. Rose, also distinguished from the former by the fact that it loses its lustre in the blowpipe flame, becomes dark-coloured, and yields, even in the flame of a spirit-lamp, the reaction of osmium; viz., that the flame is rendered luminous, as if from the burning of olefiant gas.

Fused with nitre, it yields more fumes of osmic acid than the former.

Irite is infusible B. B., and also insoluble in all acids.

Fused with nitre in a matrass, osmic acid is evolved.

16. GOLD, Au.

Its occurrence in the mineral kingdom and in metallurgical products.

Gold is almost always found in nature in the metallic state, but is never pure, being combined in every case with other metals:

a. With *silver;*

b. In combination with *mercury;*

c. In combination with *palladium;*

d. In combination with *rhodium;*

e. In combination with *tellurium;*

f. In combination with *tellurium,* when a part of the tellurium is replaced by *antimony;*

g. In combination with *tellurium* and *metallic* sulphides;

h. In a mineral, the constituents of which have not yet been quantitatively determined, viz.

Phyllinglanz, from Deutsch-Pilsen, in Hungary; it contains Sb, Pb, Te, Au, and S, with traces of Se and Ag. (It is apparently related to *nagyagite*. Dana.)

i. As an accidental ingredient of certain *iron* and *copper pyrites*, *mispickels*, and *blendes*.

Among the metallurgical products, in which gold forms a principal constituent, only the gold amalgam resulting from the amalgamation of gold ores needs to be especially mentioned. Gold is also found as a secondary constituent in many of the metallurgical products from the smelting of auriferous silver ores, which have already been mentioned under silver; viz., *fine silver*, *brightened silver*, *raw lead*, *Rohstein*, *lead matt*, as well as in the *speiss* from the smelting of auriferous mispickel.

Examination for Gold.

a. General examination for gold.

When combinations of gold with other metals are fused in O. F., on coal, they give, provided the metals combined with the gold are volatile at high temperature, a coating, *e. g.*, tellurium, antimony, mercury, and lead. The mercury may also be separated in a matrass or closed tube, as has been described under silver amalgam (p. 274), and the lead may be removed by cupellation. These metals being separated, the gold, if free from non-volatile metals, is left quite pure, and may be recognized by its gold-yellow colour. If, however, it contains such metals, *e. g.*, copper, silver, platinum, palladium, rhodium, etc., they must be separated by particular methods, which will be presently described.

If the gold contains, for example, copper and silver, which can be very easily ascertained by an examination with S. Ph. in O. F. on coal, the alloy must first be cupelled with the necessary amount of test lead (*vide* quantitative gold assay). By this means the copper is entirely removed, and an alloy of silver and gold only remains. When the button is yellow, it shows that the amount of silver is inconsiderable; it is then only tested on coal with S. Ph. in O. F., by which a bead is obtained that assumes an opal-like appearance when cool (*vide*

A gold button obtained in this way will often still contain traces of silver; if desired perfectly free from silver, regard must be paid, during the parting, to the directions which will be given under the quantitative assay of alloys for gold. When the gold contains metals which by themselves are infusible B. B., such as platinum, iridium, palladium, and rhodium, the alloy is fusible B. B. with more difficulty than pure gold; they can also be recognized by the fact that, when such an alloy is dissolved in aqua regia, any iridium remains behind, and the solution possesses a darker colour than that from pure gold. The separation of the above-mentioned metals from the gold will be specially described under the quantitative gold assay.

In order to find a small amount of gold in pyrite, arsenopyrite and chalcopyrite, as also in the various metallurgical products consisting of metallic sulphides and arsenides, the method must be followed, which will be especially described under the quantitative assay for gold in the above ores and products.

b. Blowpipe characteristics of minerals containing gold.

Native gold fuses on coal to a globule, which has a bright surface when cold. The colour of the globule appears purer gold-yellow the less silver there is present.

With S. Ph. on coal in O. F. a bead is obtained, which opalesces when cool, owing to the oxide of silver dissolved.

Gold amalgam heated in a matrass yields metallic mercury and leaves behind a spongy mass, which melted on coal with borax, unites to a gold button, containing a small amount of silver, and having therefore a pale gold-yellow colour.

Palladium-gold and *Rhodium-gold.*—Of the behaviour of these two minerals B. B. it is only known that they fuse on coal and form a malleable alloy. For the treatment of such alloys, *vide* general examination for gold.

GOLD IN COMBIANTION WITH TELLURIUM.

Sylvanite.—According to Berzelius, it gives in an open tube white fumes, which, near the assay, are gray from the sublimed tellurium. When the flame is directed upon the sublimate, the latter melts to clear, transparent drops; it smells acid, but without the least trace of a horseradish odour.

On coal it fuses to a dark gray metallic globule, and coats the coal with white fumes, which disappear with a bluish-green flame when touched by the R. F. After continued treatment, a light yellow metallic button remains, which glows just before it solidifies. The perfectly bright button when cool is malleable.

Müllerite (*sylvanite*) behaves in the open tube like the preceding mineral.

On coal it fuses easily to a globule, and coats the coal with the tellurium fumes, which, touched by the R. F., are driven off, imparting a bluish-green tinge to the flame; then, after further treatment, a yellow coating of oxide of lead is formed, and finally there remains a metallic button, which has the colour of silver, but which cannot be dissolved in nitric acid. When fused with twice its weight of chemically pure silver, and again treated with nitric acid, all the silver is dissolved, and a considerable amount of gold remains.

Phyllinglanz.—In a closed tube, at a gentle heat, the mineral is not altered; with a stronger heat it fuses and gives a trifling white sublimate, which, however, cannot be again volatilized.

In the open tube with a gentle heat, it fuses only imperfectly, but evolves sulphurous acid and a white smoke, which coats the glass tolerably near the assay, has the appearance of a combination of oxide of antimony and antimonic acid, and increases with a stronger heat, by which the mineral is fused to a globule. The fused assay is surrounded by a brown glass, which, on the edges, is translucent and of a yellow colour.

Alone on coal it fuses very easily, spreads out and volatilizes, leaving a few very small scattered gold buttons, and affords a scarcely perceptible odour of selenium. A white coat is also formed, which disappears, when touched with the flame, with a greenish-blue tinge, leaving behind yellow spots. The white coat accordingly consists principally of a mixture of oxide of antimony and sulphate of lead.

Treated with borax on coal in O. F., a white antimony coat is formed, which has, however, a dark yellow border; thus proving that some tellurium is present. The remain[illegible]etallic button possesses the lustre and colour of pure go[illegible] borax bead appears colourless and is perfectly transp[illegible].

c. Examination for gold in metallurgical products.

All that is to be noticed in the examination for gold in metallurgical products can be found under the general examination for gold.

17. TITANIUM, Ti.

Its occurrence in the mineral kingdom and in metallurgical products.

Titanium is found in nature only in the oxidized state:

a. As *acid*, alone;

b. As *acid* in combination with *earths* and *metallic oxides*.

Titanium occurs in metallurgical products:

a. In combination with cyanogen and nitrogen, partly crystallized, in certain blast-furnace slags, as small copper-red cubes, which, according to Wöhler, are composed of Ti C^2 N + 3 Ti^3 N, with 78 Ti, 18·1 N, and 3·4 C, or 16·2 cyanide of titanium and 83·8 nitride of titanium, and partly in more or less adherent, irregularly-shaped bodies in the bears, which form when blowing out iron blast-furnaces.

b. According to Kersten, certain blast-furnace slags also owe their fine blue colour to a small amount of oxide of titanium.

Examination for Titanium.

a. General examination for titanium.

Minerals consisting principally of titanic acid can be very easily recognized from their behaviour with the fluxes and soda (*vide* titanic acid). When, however, the titanic acid is combined with bases, or in general, with earths or metallic oxides, it is not always possible to recognize it immediately by means of the fluxes, because certain of the basic constituents hide the reaction of titanium. When, besides titanic acid, only the oxides of iron are present in moderate quantities, the presence of the former can be proved by its behaviour with S. Ph. in R. F., if it is known that tungsten is not present; the bead becomes dark on cooling, and more or less brownish-red. If the amount of titanium is large, a violet colour can be obtained in S. Ph., when treated in R. F. on coal with tin.

According to Riley, it is better to use a piece of metallic zinc instead of tin, when the amount of titanium in the substance is very small and when the violet colour does not make its appearance distinctly.

In complex substances, which give no decisive reaction for titanium with the fluxes, a small amount of the same may be detected in the following way:—The finely-pulverized substance is fused in a platinum spoon at a moderate red heat with six to eight times its weight of bisulphate of potassa, the mixture being melted in small portions at a time. The mass is then dissolved in just sufficient water in a porcelain dish over the lamp, and the insoluble parts allowed to settle. The solution, if concentrated, may be heated to boiling.

The clear solution is then poured into a larger dish, mixed with a few drops of nitric acid, diluted with at least six times as much water, and then boiled. If the substance contained titanium, the latter is dissolved by the fusion with bisulphate of potassa and treatment with water, but is precipitated from the acid solution by continued boiling as white titanic acid. If the solution is not acidified with nitric acid before boiling, a yellow, ferruginous titanic acid is obtained, when the substance contains iron. The precipitated titanic acid is collected upon a small filter, washed with water containing nitric acid, and tested with S. Ph., either on platinum wire or coal. If the amount of titanic acid is so small that in R. F. it does not impart to the S. Ph. the violet colour of sesquioxide of titanium, it is only necessary to add a little sesquioxide of iron, when the assay is upon a wire, or a small piece of iron wire when on coal, and to fuse the glass for a short time with the R. F.; it then appears yellowish while hot and brownish-red when cool.

If compounds containing zirconia with titanic acid are fused with bisulphate of potassa, and the mass treated with water, a part of the titanic acid is apt to remain undissolved with the zirconia: of this extended mention has been made under yttria.

b. *Blowpipe characteristics of minerals containing titanium.*

Octahedrite, *brookite* (*arkansite*), and *rutile* are infusible B. B. In the fluxes and with soda they behave like titanic acid (*vide* Tables). It need only be remarked that they dissolve in S. Ph. with more difficulty than the pure artificial titanic acid, and also that the colours produced by titanic acid in borax and S. Ph. have sometimes a different appearance, owing to a small admixture of other metallic oxides, as iron, and that with soda and nitre a reaction of manganese is often obtained.

c. Behaviour of titanium occurring in metallurgical products.

The titanium which, combined partly with cyanogen and partly with nitrogen, is found crystallized in blast-furnace slags, or only as an admixture in bears, is dissolved in borax with difficulty, but with moderate ease in S. Ph. Even from the purest portions no violet colour is obtained by dissolving it in S. Ph. and treating in R. F. The cold glass always has a more or less intense brownish-red colour, owing to the presence of iron. Even on treating the glass with tin, no violet colour is produced.

18. TANTALUM, Ta, AND NIOBIUM (COLUMBIUM), Nb.

These metals occur only as acids combined with bases, and are found in the following minerals:—

Tantalite, columbite (*vide* iron);

Pyrochlore (*vide* lime);

Yttrotantalite, hielmite, fergusonite, samarskite, tyrite, æschynite, euxenite, polycrase (*vide* yttria);

Wöhlerite, eucolite (*vide* zirconia);

Wolframite (*vide* iron);

Cassiterite (certain varieties) (*vide* tin).

Examination for Tantalum and Niobium.*

The most certain method of recognizing these metals in very complicated combinations is the same as that already given under yttria. A sufficient amount of the very finely powdered mineral is fused with bisulphate of potassa, and the fused mass dissolved in water, after being pulverized. If the mineral contains one of the above acids, with perhaps tungstic acid also, these are separated by the treatment with water, while any titanic acid present dissolves, together with the bases. The residue may be either fused with carbonate of potassa, or if free from titanic acid and zirconia, treated at once with sulphide of ammonium, to separate the tungstic acid and oxide of tin. After filtration and thorough washing, the residue is treated on the filter with dilute hydrochloric acid, to remove traces of iron, and is then examined for tantalic or niobic acids.

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* It has been definitely settled that the metal *dianium*, which von Kobell thought he had discovered in certain minerals (viz., samarskite, euxenite, etc.), does not exist, and it is therefore here omitted, although retained in the German edition. As regards the recent investigations of the acids of tantalum and niobium, see the end of this article.

The examination may be made either with the glass fluxes and cobalt solution, or with hydrochloric and sulphuric acids, and the addition of metallic tin or zinc. Rammelsberg gives the following distinctive reactions of tantalic and niobic acids.

If hydrochloric acid and zinc are added to a solution of a tantalate of an alkali, no coloration results, unless a large amount of acid is added, when there ensues a feeble bluish coloration. If niobate of an alkali is similarly treated with hydrochloric or sulphuric acid and then with zinc, the separating niobic acid rapidly assumes a fine blue colour, which gradually becomes paler, and then brown; the fluid is then also brown, and yields a brown precipitate with ammonia.

B. B. tantalic acid yields colourless glasses. B. B. niobic acid, obtained by decomposition of the chloride, yields with S. Ph. in O. F. a colourless glass, which in R. F. is violet or blue (with larger quantities).

The examination for the respective acids may also be made after fusing the substance in fine powder with carbonate of potassa. Titanic acid has no injurious effect, because the resulting titanate of potassa is but slightly soluble in water.

The fusion is performed either with separate portions, upon a stout platinum wire, or more conveniently in a small platinum crucible of the same shape as the clay crucible (Fig. 23), which is set in a square coal and heated as strongly as possible, as is described under the quantitative assay for lead. Five or six volumes of carbonate of potassa are employed, with the addition of one volume of nitre, when the bases are at a low stage of oxidation. The fused mass is treated with hot water, the solution (which is almost always coloured more or less green by manganese) filtered off, and treated with hydrochloric acid. The precipitate of the metallic acid, which is generally of a reddish tinge, owing to the presence of some manganese, is filtered out, treated with sulphide of ammonium if tungstic and molybdic acids are present, and the examination then conducted as above.

Tantalic acid may be further recognized by the following reaction. The doubtful acid which remains after the fusion with bisulphate of potassa, is mixed with five volumes of carbonate of potassa and fused, either in the platinum crucible, as above, or on platinum wire, to a clear mass, and this is dissolved in hot water, affording a clear solution, from which the respective metallic acids are thrown down by hydrochloric acid. Upon adding tincture of galls to the solution, the white precipitate assumes a light yellow colour if consisting of tantalic acid, while in presence of niobic acid it becomes orange-yellow or orange-red.

If one of the compounds here included contains silicic acid, the examination is conducted as above described (see zirconia), for the decomposition of wöhlerite and eudialyte, which can be decomposed by hydrochloric acid.

Should the compound not be decomposed by hydrochloric acid, it must be fused with soda and borax, the fused mass evaporated to dryness with hydrochloric acid, and then treated with water. The residue of acids is then to be washed with acidulated water on a filter, dried and fused with five volumes of carbonate of potassa, as before; the beads are pulverized and further treated, as above. The reactions for the acids in question are in no way injured by the presence of the silicic acid.

[NOTE.—In the foregoing pages, as well as in the tables (p. 100, *et seq.*), the old formulas for the acids of tantalum and niobium have been retained; the recent investigations of Marignac and Blomstrand, however, indicate that there are only two acids, tantalic acid and niobic acid, and that H. Rose's hyponiobic acid is identical with niobic acid. The formulas for the acids are $Nb\,O^5$ and $Ta\,O^5$; Marignac gives the equivalent of niobium as 94 ($H=1$), and the equivalent of tantalum is given as 182. Transl.]

19. ANTIMONY, Sb.

Its occurrence in the mineral kingdom and in metallurgical products.

This metal is found in nature—

a. In the *metallic state*, alone;

b. In combination with *other metals;*

c. In combination with *sulphur*, alone and also with other *metallic sulphides ;*

d. In a combination of *sulphide and oxide of antimony;*

e. In the *oxidized state ;*

f. In combination with *earths* and *metallic oxides.*

In metallurgical products antimony only forms an essential ingredient in the sulphide of antimony, $\overset{''}{\mathrm{S}}\mathrm{b}$ extracted by liquation; as a secondary constituent it is found in several products from lead and silver smelting, when the smelted or amalgamated ores were stibiferous silver or lead ores, or were not free from an admixture of stibnite. Here are to be specially mentioned:

Raw lead and lead reduced from abstrich (*vide* lead);

Retort-silver (*vide* silver);

Raw matt, lead matt, copper matt, furnace crust (*vide* iron), in which products the antimony is present as $\overset{'''}{\mathrm{S}}\mathrm{b}$; also abzug and abstrich (*vide* lead), which products contain the antimony as antimonic acid Sb combined with oxide of lead.

Besides these products there are several others, which sometimes contain small quantities of antimony, as certain kinds of black copper and speiss, and the litharge that is obtained from the cupellation of lead containing antimony.

Examination for Antimony.

a. General examination for antimony.

The examination for antimony is very simple, and is often limited to a test of the substance on coal or in an open tube.

Alloys are generally tested on coal, and the antimony recognized by the coat which it deposits (*vide* behaviour of antimony on coal).

If the antimony is combined with metals which also give a coating on coal, a coating of oxide of antimony is formed, it is true, that is visible when the amount of antimony is not too small, but the usually less volatile coating, that is at the same time formed by the other metals, appears sometimes changed in colour, as is the case with lead that contains antimony. If, however, such a combination is treated with a little vitrified boracic acid, so that the fused glass is covered with the blue flame, the metallic button remaining on the side of the glass, the lead is dissolved by the boracic acid as oxide, and a coating of pure oxide of antimony is formed, if too much heat is not applied. The method is the same for bismuth.

If the antimony is combined in small amounts with metals, such as copper, from which it can only be separated with difficulty, no coating is formed by a slow volatilization; such an alloy is treated on coal in O. F. with S. Ph., until a part of the antimony has been oxidized and taken up by the glass, which is then separated from the metallic button and treated in R. F. on another part of the coal with tin. It is then observed whether, upon cooling, it assumes an opaque dark-gray or black colour, from which the presence of antimony may, in most cases, be distinctly recognized (see Tables). Since, however, a portion of the metals combined with the antimony, if they are oxidizable, are also oxidized and taken up by the glass, it must be considered what colours these oxides impart to the glass when treated with tin. Especial regard must be paid to this point when bismuth is present, for its oxide behaves in S. Ph. almost the same as the oxides of antimony. If, therefore, both metals are present together, combined with a third metal, the examination in S. Ph. is not decisive; in this case a larger assay-piece must be used, and the point decided by examining the coatings very carefully, or the wet way must be resorted to. In the latter case the alloy is dissolved in aqua regia, the solution mixed with ammonia in excess, and the metals precipitated as sulphides by sulphide of ammonium, in which some sulphur has been previously dissolved. The precipitated sulphide of antimony is then redissolved by heating, the solution diluted with water, filtered, and the sulphide of antimony precipitated by dilute hydrochloric acid and collected on a filter. When dry it is either tested on coal or in an open tube.

In the examination for copper it was observed, that when a bead of S. Ph. containing oxide of copper is treated for a short time on coal with tin, it becomes opaque and red, but afterward quickly dark gray to black, if it contained even a small quantity of antimony. As, then, a bead of this kind becomes red when cool only after a long-continued treatment in the R. F., a small amount of antimony, even in copper, can be easily detected by an examination with S. Ph.

Metallic sulphides are examined for antimony partly on coal, partly in an open tube.

According as the substance is rich or poor in sulphide of antimony, there are formed in the tube, besides sulphurous acid, more or less antimonial fumes, which are deposited in the form of powder upon the glass, partly as oxide of antimony, and partly as a combination of oxide of antimony and antimonic acid. The former is furthest from the assay and can be driven from place to place by heating; a part of it, however, is apt to be oxidized, at the expense of the air that flows through the tube, to a combination with antimonic acid, which, like that deposited upon the under side of the glass near the assay, can neither be volatilized nor fused. When the amount of antimony is inconsiderable, the combination of the two oxides is almost exclusively formed.

If the substance contains sulphide of lead, as well as sulphide of antimony, thick fumes are evolved, but only the smallest part of the same is deposited as volatile oxide of antimony. It is changed for the most part to a combination of oxide of antimony and antimonic acid, which is mixed with sulphate of lead, and near the assay with antimonate of lead, and which cannot be volatilized.

If the substance contains arsenic, it is observed that when the assay is only heated a short time, a mixture of pulverulent oxide of antimony and crystalline arsenous acid is deposited upon the glass. If a mixture of sulphide of arsenic and sulphide of antimony, in which the quantity of the latter is small, is to be examined, it is put into a closed tube and the sulphide of arsenic sublimed by a gentle heat, the greater part of the sulphide of antimony remaining behind and having a black colour. The lower part of the tube is then cut off, and the sulphide of antimony taken out and tested in an open tube. As a perfectly decisive result is obtained in this way, this test is recommended for the examination of the mixture of the sulphides of arsenic and antimony, which, in qualitative analysis of compound substances in the wet way, is separated by hydrosulphuric acid, or precipitated by acids from a solution of sulphide of ammonium. It must, however, be previously dried thoroughly.

The examination of metallic sulphides for antimony can also take place on coal, because the coating of oxide of antimony can be easily distinguished from others that are similar.

If the substance contains arsenic, this is first volatilized by a weak flame, provided it is present in not too small an amount and not combined with nickel or cobalt; the coal, far distant from the assay, is covered with a white coating, which is grayish in the thinner parts. If no arsenic is present, a thin coat of antimony is often deposited. When no more arsenical fumes are observed, the coat of arsenous acid is driven off by a gentle flame, without igniting the coal, in order to obtain a clean surface upon which the antimony coat, formed by a further heating of the assay, can be recognized by its behaviour when touched with the R. F. If, however, the substance contains lead or bismuth, a white coat is formed, which consists of a mixture of oxide of antimony and sulphate of lead or bismuth; near the assay a yellow coat of oxide of lead or bismuth is also formed, which, however, contains antimony. The method for detecting with certainty the presence of antimony in such substances has been specially described in the general examination for lead. The method described there refers principally to substances containing lead; it can, however, be employed also for substances that contain bismuth.

When the substance contains zinc, there is also deposited on the coal, near the assay, a coat of oxide of zinc, which, however, can be easily distinguished from an antimony coat by not being volatilized in the outer flame, while the oxide of antimony can be driven from place to place, or almost entirely volatilized.

Substances which contain antimony as oxide are best tested on coal, either by themselves, or still better in the R. F., with the addition of soda; the antimony is reduced, volatilized, and again oxidized, forming a coat on the coal. When oxide of lead is to be tested, a part of which is combined with antimonic acid, abstrich for example, the blast must not be too continuous, or else there will be too much lead driven off, by which the antimony coat is rendered indistinct.

If the antimony is mixed or combined with metallic oxides, such as those of tin or copper, which when reduced retain the antimony, they are treated on coal in R. F. with soda and borax; by this means small, easily fusible, metallic globules are separated, which, when the amount of antimony is inconsiderable, give very little or no oxide of antimony on coal, even when treated in the R. F. for a long time. The metallic globules are separated by triturating and washing the fused mass in a mortar with water, then fused on coal in R. F. with three to five times their volume of test lead and a little vitrified boracic acid. If the glass only is exposed to the R. F., the antimony is volatilized and coats the coal distinctly with its oxide.

b. Blowpipe characteristics of the minerals that contain antimony.

NATIVE ANTIMONY

Behaves B. B. on coal like pure antimony; it gives forth sometimes, however, a distinct odour of arsenic, and when volatilized in R. F. with borax, gives a more or less distinct reaction of iron.

A small silver button is usually obtained when it is fused with test lead, the metallic globule treated in O. F. until all the antimony is driven off, and the lead then finally cupelled.

ALLEMONTITE

Fuses very easily, volatilizes with a strong odour of arsenic, and coats the coal with arsenous acid and oxide of antimony.

ANTIMONY IN COMBINATION WITH SULPHUR AND OTHER METALLIC SULPHIDES.

Stibnite fuses very easily when moderately heated in a closed tube, and sometimes yields a small sublimate of sulphur; more strongly heated with the blowpipe flame, a sublimate is formed, which when quite cool appears cherry-red to brownish-red.

Heated in an open tube it fuses very easily, and evolves sulphurous acid and dense antimony fumes, the greater part of which is deposited on the glass near the assay as a combination of oxide of antimony and antimonic acid; a portion, as oxide of antimony, floats through the tube and escapes, or is partly deposited upon the glass. When too strongly heated, the coat is apt to be tinged with red.

On coal, fuses easily, spreads out, partly sinks into the coal and is partly volatilized; the portion in the coal emerges again, on continued blowing, in the form of small, bright globules, which are then volatilized. During the operation sulphurous acid is disengaged and a coat of oxide of antimony formed.

Berthierite heated in a closed tube, decrepitates sometimes, fuses, yields a small sublimate of sulphur, and by stronger heat a black sublimate of sulphide of antimony, which becomes cherry-red when cool.

In the open tube behaves like stibnite.

Fuses easily on coal, and leaves, when the antimony is driven off, a black, slag-like, magnetic mass, which gives the reactions for iron, and, in the variety at least from Bräunsdorf, a manganese reaction with soda and nitre.

SULPHIDE OF ANTIMONY IN COMBINATION WITH OXIDE OF ANTIMONY.

Kermesite in a closed tube fuses very easily, gives at first a little oxide of antimony, and then a slight, yellowish-red sublimate. With stronger heat it boils and yields a sublimate of sulphide of antimony. In the open tube and on coal behaves like stibnite.

OXIDE OF ANTIMONY.

Valentinite and *Senarmontite* in the matrass fuse and partly sublime.

On coal, fuse very easily and coat the coal with oxide of antimony. In R. F. a part is reduced to metallic antimony, which, however, is volatilized by continued blowing. When the R. F. is directed upon the assay, the outer flame is coloured pale greenish-blue.

Cervantite is gradually reduced when treated on coal in R. F.; the reduced antimony is volatilized, covering the coal with oxide and leaving behind any admixtures of foreign substances which are not volatile. With soda it is quickly reduced, so that small globules of antimony are separated, which disappear, however, on further treatment.

Certain antimony ochres yield water in the matrass.

a. Examination for antimony in metallurgical products.

The method of detecting the presence of antimony in the abovementioned metallurgical products is given partly in the examination for iron, partly under lead, and also under silver.

20. TUNGSTEN, W.

Its occurrence in the mineral kingdom and in metallurgical products.

Tungsten occurs in nature only as an acid:

a. Alone;

b. In combination with *bases.*

In metallurgical products tungsten is sometimes found in small quantities in *steel,* in certain kinds of *tin;* more frequently and in larger amounts in the furnace deposits, and in *scraps* from the refining of tin; principally, however, in certain tin slags as acid.

Examination for Tungsten.

Tungstite acts before the blowpipe, according to von Kobell, as follows:

On coal, in R. F., becomes black.

In S. Ph. in O. F. it dissolves to a colourless or yellowish glass, which in R. F. becomes fine blue when cold; it behaves otherwise like pure tungstic acid (see Tables).

In the other above-mentioned minerals, the presence of tungstic acid, if not in too small quantities, can be easily recognized by the examination with S. Ph.; the bead in R. F. becomes blue when cold, or, in the presence of iron, more or less red. Since, however, substances that contain the oxides of titanium and iron behave in the same way as those in which tungsten and iron are present, it is advisable, in certain cases, to make a special examination for tungsten.

Upon the decomposition of complex substances, such as compounds of tantalic, niobic, and titanic acids, by fusion with bisulphate of potassa and a further treatment, partly in the dry, and partly in the wet way (see yttria), the tungsten is separated as sulphide. It is then only necessary to ignite this on coal in O. F., thereby changing it to oxide, and to test it on a platinum wire with S. Ph. If enough has been dissolved, the glass appears, when treated in O. F., yellowish while hot, but becomes colourless when cold; after treating it a short time in R. F., it becomes blue when cold, and bluish-green by a longer treatment in the same.

If it is required to prove the presence of tungstic acid in substances in which it is uncertain, from their behaviour with S. Ph., whether they contain tungstic or titanic acid, they must be treated in the following way: the finely pulverized substance is mixed with five times its volume of soda, the mixture made into a sort of paste with a little water, and portions of this melted in O. F. on platinum wire. Whenever the latter becomes full, the fluid mass is shaken off into a porcelain dish. The fused mass is now pulverized and boiled with water in a deep porcelain dish, in order to decompose, by the reducing action of the residue, any manganate of soda that might have been formed and dissolved.

The solution is then poured off into another dish, as soon as it has become clear by the settling of the insoluble earths and metallic oxides, to which latter class titanic acid belongs. The clear solution, which contains tungstate of soda, is mixed with a few drops of nitric acid, until it gives an acid reaction, the tungstic acid separating as a white powder, which becomes dense and lemon-yellow when the whole is heated nearly to boiling. In this operation especial care must be taken not to have the solution of tungstate of soda, which also contains carbonate of soda, too concentrated, because the tungstic acid, which has been separated by nitric acid, does not turn yellow in very concentrated solutions. The acid can then be further examined with S. Ph.

It must also be noticed here, that if substances containing at the same time much tantalic or niobic acids are tested in this way for tungstic acid, the solution contains these acids also in combination with soda, and that, by the addition of nitric acid, a white precipitate is formed, which, when the solution is heated, does not turn distinctly yellow, and sometimes not at all so. To recognize the tungstic acid in this case, the precipitate must be digested with sulphide of ammonium, and the tungsten dissolved out as sulphide, which is further treated as described above.

When scheelite (tungstate of lime) is finely powdered and digested in a test tube with hydrochloric acid, the tungstic acid separates as a yellow powder; if a small piece of metallic tin is now added, the whole is coloured blue by the formation of tungstate of tungsten. When further heated, the colour becomes gradually darker. This test can also be used directly in the examination of wolframite. A portion of the finely-pulverized mineral is digested with hydrochloric acid, until a moderately concentrated solution is obtained; this, when clear, is poured off from the residue, a piece of tin is added and heat applied, when the solution generally assumes, in a short time, a blue colour.

If salts of tungsten occur in combination with silicates, as is the case in certain tin slags, the tungstic acid remains mixed with the silica, when the above compounds are decomposed into their separate constituents. Its presence, if the amount is not too small, can be proved by fusing the mixture in O. F., as described above for tantalic acid containing tungsten, with a bead of S. Ph. that contains iron, and treating the glass for a while in R. F., observing whether the bead assumes, when cold, a dark yellow to red colour. The silica, which remains almost entirely undissolved, does not affect this reaction. If, however, the amount of tungstic acid in proportion to the silica is very small, no reliable result is obtained by this test. If, in the examination of a tin slag, which always contains protoxide of iron, with S. Ph., no really distinct reaction for tungstic acid has been observed, the silica which has been separated by the decomposition of the slag, can be treated on the filter with sulphide of ammonium, as described under yttria, in the separation of tungstic acid from tantalic and niobic acids. The sulphide of tungsten in solution is precipitated by dilute hydrochloric acid, and tested as described above.

The presence of tungstic acid in tin slags may also be frequently recognized by the deep indigo-blue solution, which is formed when the pulverized slag is warmed in a test tube with hydrochloric acid. This is due to the fact that the tin slags contain, besides oxide of tin, finely-divided metallic tin, which, in dissolving, acts as a reducing agent upon the tungstic acid in the solution of the bases, and forms tungstate of tungsten, which colours the solution blue.*

The examination of metallic tin for tungsten has already been described in the general examination for tin.

21. MOLYBDENUM, Mo.

Its occurrence in the mineral kingdom and in metallurgical products.

Molybdenum occurs in nature:

a. In combination with *sulphur ;*

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* If tin slags, which have been freed from the particles of metallic tin by careful washing, behave in the same way, it is owing to the fact that it is not possible, in these slags, to separate mechanically all the finely-divided tin from the powdered slag.

b. As *acid* by itself;

c. In combination with *oxide of lead.*

Molybdenum is also found in small quantities in a few products from copper and tin smelting; viz.—

a. Metallic in certain kinds of *raw copper, refined copper,* and *tin,* also in certain *bears,* which are sometimes deposited upon the sole of the furnace when smelting, in shaft furnaces, copper and tin ores that contain molybdenum. Finally, it is also found

b. As *oxide* in the various scraps and slags which are produced in smelting molybdiferous copper and tin ores, as well as in refining raw or black copper, and in the purification of tin.

Examination for Molybdenum,

Including the blowpipe characteristics of the minerals belonging here.

Molybdenite in a closed tube remains unaltered. In the open tube yields only sulphurous acid.

When a thin scale of the mineral is heated in the forceps with the point of the blue flame, no fusion takes place, but a yellowish-green streak is seen in the middle of the outer flame.

On coal in O. F. the mineral disengages a smell of sulphurous acid and coats the coal, when the assay is held as far as possible from the flame, with crystalline molybdic acid, which appears yellowish when hot, and becomes white upon cooling; near the assay the characteristic copper-red film of binoxide of molybdenum with its metallic lustre is observed. The assay diminishes in volume, but does not fuse.

Heated in a platinum spoon with nitre, it deflagrates with a flash and dissolves in the fused salt, leaving, however, yellow flakes, which remain undissolved when the mass is treated with water, and which behave in the fluxes like sesquioxide of iron. In the alkaline solution, the molybdic acid which is formed can be easily detected, as described further on.

Molybdite behaves on coal by itself like molybdic acid. Treated with soda, it sinks into the coal, leaving some iron behind.

In substances which do not contain too little molybdenum, this metal can be found in the following way:—The substance to be tested is finely pulverized and fused, if it contains the molybdenum as sulphide, with three times its volume of nitre, but if it contains the metal as acid, with a mixture of nitre and soda, in the platinum spoon, until all the molybdenum is combined as acid with the alkaline bases present. The fused mass is dissolved in water in a small porcelain dish over the lamp, the clear solution poured off from the residue into a porcelain capsule, and mixed with a few drops of hydrochloric acid, until it gives a feeble acid reaction. It is then warmed, and a small piece of bright copper foil added; in a short time it is quickly coloured; commencing from the spot where the foil lies, fine dark-blue, by the separated molybdate of molybdenum. Tungstic acid, treated in this way, assumes only gradually a feeble blue colour.

According to von Kobell, molybdic acid can also be recognized in its combinations by heating the finely pulverized substance in a porcelain dish with concentrated sulphuric acid, and then adding alcohol. The fluid when cold acquires, especially upon the sides of the dish, a fine azure-blue colour.

In order to find a small amount of molybdenum in the above-mentioned *metallurgical products*, it is necessary to have recourse to the wet way. A moderate quantity of the products consisting of combinations of metals is dissolved in aqua regia (products containing tin preferably in nitric acid), diluted with water, filtered, the solution mixed with ammonia in excess, sulphide of ammonium added, and the glass covered and put in a warm place, to allow the precipitated sulphides to settle. The sulphide of molybdenum which remains in solution, is precipitated, after filtration, by very dilute nitric acid, washed with water, to which a little sulphide of ammonium has been added, and tested when dry B. B. It behaves in general, when pure, like molybdenite.

Scraps and *slags*, which contain the metals present as oxides, are pulverized and fused with bisulphate of potassa, the melted mass dissolved in water, filtered, and the molybdic acid separated from the metallic oxides by ammonia and sulphide of ammonium, as described above.

22. VANADIUM, V.

Its occurrence in the mineral kingdom.

Vanadium belongs to the rare metals; it has been found, up to the present time, only as acid combined with bases in the following minerals:

Descloizite,
Dechenite,
Vanadinite,
Eusynchite,
Aræoxene,
Chileite,
vide lead;

Volborthite,
Vanadate of lime and copper (Kalkvolborthit),
vide copper.

Furthermore, it forms an unessential ingredient in:
Conichalcite (*vide* copper), and in
Hydrophite (*vide* magnesia).

It has also been found in very small quantities in certain clays and iron ores, in uraninite, in cupriferous slate (Kupferschiefer) and in slags.

Examination for Vanadium.

If the amount of vanadium in any substance is not too small, it can be recognized by testing with borax or S. Ph., provided the reaction is not suppressed by other colouring metallic oxides. The fluxes appear yellow after treatment in O. F., but change their colour in R. F., appearing brownish while hot, and emerald-green upon cooling (*vide* vanadic acid).

If substances containing vanadic acid, but free from combinations of silica, are finely pulverized and fused in a platinum spoon with twice their volume of soda and the same amount of nitre, and the melted mass then treated with water, the vanadate, nitrite, and excess of nitrate of potassa and carbonate of soda added are dissolved, the other bases remaining behind, provided they have not also combined as acids with a part of the alkalies. When, therefore, the substance contains at the same time chromic, phosphoric, arsenic, or sulphuric acids, these are also brought into solution, combined with potassa and soda. The solution of the alkaline salts, after it has become clear, is decanted off from the residue into a porcelain dish, saturated with acetic acid and a small quantity of crystallized or pulverized acetate of lead added, which dissolves without producing cloudiness, if the solution is free from those acids which decompose this salt and form a precipitate. If, however, vanadic acid is present, neutral vanadate of lead is formed, which, when strongly heated, falls to the bottom and assumes a pale yellow colour. The behaviour of this salt in borax and S. Ph. establishes the presence of vanadium. If the solution contains chromic acid also, the precipitate becomes proportionably darker yellow, but paler in the presence of phosphoric, arsenic, or sulphuric acid; in the latter case it is necessary, before the precipitate can be tested with borax or S. Ph., to collect it on a filter and treat it on coal in R. F., until all the arsenic and the greater part of the lead are volatilized. When chromic acid is present in the precipitate, the bead of S. Ph., treated in a pure O. F., does not appear yellow, but yellowish-green.

According to von Kobell, the combinations of vanadic acid with lead can be recognized by the following reaction. The solution of the same in concentrated hydrochloric acid (by which a great deal of chloride of lead is separated), when concentrated, after an admixture of alcohol, and decanted from the chloride of lead, assumes an azure-blue colour, upon the addition of water. Chromate of lead treated in this way also yields a green solution, which, however, remains green.

If a substance contains, together with vanadium, a small amount of iron, the latter may be best detected by fusing the substance with three to four parts by weight of bisulphate of potassa, dissolving the mass in water, and testing the clear solution with a solution of ferrocyanide of potassium. When the solution is free from iron, a green, flocculent precipitate of ferrocyanide of vanadium is formed; if it contains iron, however, this is present as sesquioxide, and imparts to the solution the dark blue colour of Prussian blue.

Silicates, such as slags, are decomposed by a fusion on coal

with soda and borax; the mass is then pulverized and treated in a platinum spoon with an equal volume of nitre, and then further treated as has been described above.

23. CHROMIUM, Cr.

Its occurrence in the mineral kingdom.

It occurs:

a. Metallic, in very small quantities in
Meteoric iron (*vide* iron).

b. As *oxide* in
Chromite (*vide* iron);
Chromic ochre,
Miloschite, } *vide* alumina;
Serpentine,
Emerald (*vide* glucina).

Also in very small amounts in the following aluminates:
Spinel, ruby (*vide* magnesia);
Chrysoberyl, green (*vide* glucina).

c. As *chromic acid* in
Vauquelinite,
Crocoite,
Phœnicochroite, } *vide* lead.

Examination for Chromium,

Including the blowpipe characteristics of the minerals belonging here.

a. General examination for chromium.

Minerals in which the sesquioxide of chromium or chromic acid forms an essential ingredient, afford in most cases a distinct reaction of chromium, when they are tested in the O. F. with borax or S. Ph.; the beads appearing yellowish-green when perfectly cool (*vide* sesquioxide of chromium). In R. F. the green colour, if the substance is free from oxides of lead or copper, becomes finer, and even, with a large amount of chromium, a pure emerald-green. If, however, these metallic oxides are present, the beads become, when cool, opaque red or gray, the green colour of the chromium being hidden.

A small amount of iron in minerals containing chromium is easily detected, in the same way as in minerals containing vanadium, by fusing with bisulphate of potassa, dissolving in water, and testing the solution with ferrocyanide of potassium.

The minerals which are coloured blood-red by sesquioxide of chromium, viz. pyrope and the spinel from Ceylon, have this peculiarity, that, by simple heating in the forceps, they become black and opaque; on cooling, however, yellowish, or chrome-green, then almost colourless, and finally just as red as before heating. Those silicates which are coloured red by chromium and iron together, become also opaque by heating, but assume immediately, while cooling, their original red colour and transparency.

Minerals which contain little sesquioxide of chromium, but other colouring oxides of metals in larger quantities, and which give no satisfactory reaction of chromium in borax or S. Ph., can be examined for chromium, the silicates and spinel excepted, in the following way:

A small quantity of the mineral is pulverized as finely as possible, mixed with twice its volume of soda and the same amount of nitre, and the mixture fused with a strong O. F., in the loop of a platinum wire or in a platinum spoon, until it is thought that all the chromium has been changed to chromic acid. In this way chromates of the alkalies are formed, which are dissolved in water in a porcelain dish, with the aid of heat. If manganese is present in the mineral, manganates of the alkalies are formed during the fusion; these impart, in the beginning, a green colour to the solution, but are decomposed by the residue of oxides when heated to boiling.* This solution is then, without separating it from the residue, strongly acidified with acetic acid and boiled. If the substance contained lead, the alkaline solution must be decanted from the residue before the acetic acid is added.

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* When the manganates of the alkalies are present in such large amounts that the oxides cannot reduce them, it is only necessary to ignite in a matrass a small piece of pure siderite, pulverize, when cool, the proto-sesquioxide of iron thus formed, add it to the assay, and heat the whole to boiling; by this means, all the manganic acid is reduced and separated from the solution.

When the acid solution is perfectly clear, it is carefully poured off from the residue into a porcelain dish, a small crystal of acetate of lead added, and the whole stirred with a glass rod. As the acetate of lead dissolves, the free chromic acid combines with the oxide of lead, forming a lemon-yellow powder, which quickly falls to the bottom, and, after filtration, gives with borax or S. Ph. in O. F. a bead that appears fine green when cool. In this manner very small quantities of chromium can be detected. If the substance contains sulphuric acid, the chromate of lead is mixed with the sulphate and the yellow colour of the former rendered proportionably paler according to the amount of the latter present. With phosphoric acid the behaviour is the same. These admixtures, however, do not affect the reaction of the chromium, when the resulting precipitate is tested with borax or S. Ph.

Silicates which contain only little chromium, but much iron or other colouring oxides of metals, and which only give with the fluxes the colours of iron or the other oxides, cannot be examined for chromium exactly according to the method just described, as silicates are not decomposed by nitre. It is therefore necessary to choose another method by which the other constituents can be found at the same time. The pulverized mineral is fused on coal in O. F. with one to one and a half times its volume of soda, and one-half to three-fourths parts of borax, to a clear bead; this is pulverized and evaporated to dryness with hydrochloric acid.

The chlorides thus formed are dissolved in water, the silica filtered off, the protochloride of iron in the solution changed to sesquichloride by boiling with a few drops of nitric acid, and the bases, viz., sesquioxide of chromium, iron, alumina, etc., precipitated by ammonia from the acid solution. The precipitate is collected on a filter, washed, and fused with soda and nitre, as above. By this means chromates of the alkalies are formed, which can be decomposed by acetic acid and acetate of lead, as described above.

Spinel is fused on coal in O. F. with two parts by volume of soda, and three parts borax, to a bead, the latter pulverized, mixed with an equal amount of nitre, and fused in a platinum spoon. The mass is now dissolved in water, and the solution acidified with acetic acid, and tested with acetate of lead to ascertain whether the spinel in question contains chromium or not. If a precipitate is formed, it must be tested with borax.

b. Blowpipe characteristics of the minerals belonging here.

Berzelius has examined B. B. various *chrome ochres,* from the departments of Saone and Loire in France, from Elfdalen and Martanberg.

The variety from France loses its colour when heated, and becomes almost white, does not fuse, but shows a slag-like surface, which, under the magnifier, appears to be made up of vitrified and unfused particles.

The sesquioxide of chromium is dissolved in borax and gives the glass a fine green colour. The piece becomes white and dissolves with difficulty. In S. Ph. it is also soluble with difficulty, and the same quantity gives a fainter colour than in borax.

It is dissolved in soda with difficulty, and requires a large amount of the flux. The glass, even when fused, is not transparent, and appears, when cool, like a dirty grayish-green enamel.

The *chrome ochre* from Elfdalen behaves like the above, as does also the clay containing chromium, from Martanberg, except that the latter, with a strong flame, fuses completely to a black slag.

Wolchonskoite from Peru behaves, according to Berzelius, as follows:

In a matrass yields water and changes its green colour to a brownish one.

In the forceps, shows upon the edges traces of melting, but does not fuse, cracks upon the surface, and becomes brown.

Borax and S. Ph. dissolve it imperfectly, giving the chromium colours. The insoluble portion is black.

With soda on coal it fuses with effervescence to a globule, which when cool appears green and yellow in spots. On platinum foil gives chromate of soda, which is fluid, and an undissolved, dark-red mass.

Rhodochrome in a matrass gives water and becomes grayish-white. In the forceps fuses only on the extreme edges to a yellow enamel.

In borax it dissolves entirely, but leaves in S. Ph. a silica skeleton, colouring the beads chrome-green.

With soda fuses to an opaque yellow mass.

Ouvarovite (*chrome garnet*), from Bisersk in Siberia, gives water in a matrass, according to Berzelius, and becomes opaque and dirty yellow: turns green again, however, when cool.

In the forceps is infusible, but appears darker and brownish on the edges, where the heat was the strongest.

In borax it is dissolved very slowly indeed, giving a chrome-green glass. In S. Ph. also very slowly dissolved. The glass shows the usual colours of chrominm; transparent red when hot, then opaque, and when perfectly cool, clear emerald-green.

With soda on coal forms a greenish-yellow slag. On platinum foil the fluid soda is coloured yellow by the chromium.

24. ARSENIC, As.

Its occurrence in the mineral kingdom and in metallurgical products.

Arsenic is not very rare; it is found under various conditions, viz.:

a Metallic and *alone* in

Native arsenic;

Arsenical bismuth (*Arsenglanz*), consisting, according to Kersten, of 97 As and 3 Bi.

In combination with other metals, viz., manganese, iron, cobalt, nickel, copper, and antimony (*q. v.*).

b. Combined with *sulphur* in

Realgar;

Orpiment;

In combination with sulphur and other metallic sulphides, including several of the minerals enumerated under iron, cobalt, nickel, copper, silver, and antimony.

c. As *arsenous acid;*

d. As *arsenic acid* in combination with bases; viz., lime, oxide of iron, protoxides of cobalt and nickel, and oxides of lead and copper (*q. v.*).

Since many arsenical ores and minerals are worked on the large scale, either alone, or with other ores, for the metals they contain, and since arsenic can only be separated with great difficulty from certain metals by roasting, it in consequence not only forms a chief constituent of the actual arsenical products, viz., *metallic arsenic, realgar, orpiment,* and *white arsenic,* but is also a frequent ingredient of certain other products, which are to be further treated.

The latter embrace especially the products already mentioned under iron, cobalt, nickel, lead, tin, copper, silver, and gold; viz.—

Rohstein, lead and *copper matts, cadmia, abzug, abstrich,* and *lead, cobalt* and *nickel speisses.*

Examination for Arsenic,

Including the blowpipe characteristics of the minerals belonging here.

A very simple test generally suffices to indicate with certainty small quantities of arsenic, even in compound substances, provided it is not combined with nickel or cobalt, or as acid with their oxides, in which case a special method is necessary.

In addition to its volatility when heated on coal, arsenic has the peculiarity of diffusing a striking garlic odour, and coating the coal with arsenous acid; it may also be sublimed unaltered in the closed tube, collecting in a crystalline form on the glass, through which it shows a metallic lustre. The acids of arsenic can be very easily reduced to metal and recognized as such, as will be seen from the methods described below; arsenous acid is also volatile.

Native arsenic sublimes in the closed tube, sometimes leaving a fixed metallic mass, which with fluxes on coal often shows iron, cobalt, or nickel. Another portion of this mass fused on coal with enough test lead and borax in R. F. and then cupelled on bone-ash, frequently leaves a silver button.

On coal behaves like pure arsenic, but frequently leaves a slight residue of arsenic combined with Fe, Co, Ni, and often containing a little Ag.

Arsenical bismuth from the Palmbaum mine, near Marienberg, Saxony, yields at first in the closed tube sulphide of arsenic, then metallic arsenic, and leaves a trifling dark-gray residue, showing with the fluxes iron, cobalt, and bismuth.

Gently heated in the open tube, evolves sulphurous and arsenous acids; more strongly heated, yields first a little sulphide of arsenic and then metallic arsenic. Kindled B. B. on coal, it continues to burn of itself, giving off gray arsenical fumes and becoming surrounded with crystalline arsenous acid.

COMPOUNDS OF ARSENIC WITH OTHER METALS.

Some of these yield a sublimate of metallic arsenic in the closed tube; others do not (*vide* arsenides under iron, cobalt, nickel, and copper). Occasionally a little arsenous acid is formed by the inclosed air. In the open tube they all yield arsenous acid, mingled with oxide of antimony in case the compounds contain antimony. The powder will yield a sublimate when a fragment of the substance fails to do so.

Most of the metallic arsenides treated on coal in R. F. yield part of their arsenic, which volatilizes and forms a coat of arsenous acid. If there is a considerable proportion of arsenic present, the assay evolves copious grayish-white fumes, which produce the garlic odour of the suboxide, and thus at once show the presence of arsenic; but if the amount is trifling, the fumes are not always perceived, and the odour seldom during the blast. In this case the glowing assay should be held under the nose, in order that the odour of the small quantities of escaping arsenic may be perceived. When but little arsenic is combined with metals from which it is separated with difficulty, as cobalt and nickel, the compound may be fused in O. F. with test lead on coal, and the presence of volatilizing arsenic ascertained by the odour.

Should none of the preceding methods suffice to detect a little arsenic combined with a metal, or in a metallic compound, the following process will serve to detect it. Brittle metals and metallic compounds which can be pulverized are reduced to powder, or, if malleable, they are reduced by filing, and about fifty to seventy-five milligr. are then mixed in the agate mortar with five to six volumes of nitre and ignited in the platinum spoon, with the aid of the blowpipe flame, according to p. 122, until no more metallic particles are visible. The metals oxidize and arsenic acid is formed, which combines with the potassa, while nitrous acid is liberated.

The mass in the spoon is now digested with water over the lamp-flame in a porcelain dish, until everything is removed from the spoon, after which two separate methods may be employed.

By the first the clear solution is poured off from the oxides into a little porcelain vessel, acidified with hydrochloric acid, thirty to fifty milligr. of sulphate of magnesia dissolved in it with the aid of heat, an excess of ammonia added, and the whole heated to boiling. Arsenate of ammonia and magnesia separates and settles quickly when the vessel is removed from the flame. The quite clear fluid is decanted from the precipitate, which is washed by boiling it with strongly ammoniacal water, again allowed to settle, and freed by decantation from the fluid, after which it is immediately dried in the vessel. The dry salt is mixed in the mortar with three volumes of neutral oxalate of potassa, or, according to Fresenius and von Babo, with six volumes of a mixture of cyanide of potassium and soda in equal parts, and then treated on coal, or in a matrass with a narrow neck. In the former case it is fused in the R. F. and the volatilized arsenic detected by the odour; in the latter case it is at first moderately warmed over the spirit-lamp in the matrass to expel any traces of moisture, which are collected by an inserted roll of blotting-paper, after which the mixture is heated to fusion. The arsenic acid is reduced and forms a sublimate of metallic arsenic in the neck of the matrass at *a*, Fig. 66. Should the amount of arsenic be too small to produce a distinct mirror, it is only necessary to cut off the neck just above the sublimate with a file and then to hold the portion of the matrass on which the sulimate is situated in the flame. If the sublimate consists of arsenic, it will volatilize and yield the arsenic odour.

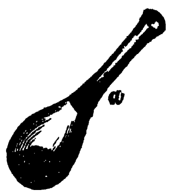

Fig. 66.

The second method consists in decanting the solution of arsenate, nitrite, and nitrate of potassa from the residue into a test tube, adding a few drops of sulphide of ammonium and agitating the whole, after which the resulting sulphide of arsenic is precipitated with dilute hydrochloric acid. The fluid is boiled to facilitate the separation of the precipitate, which is filtered out, thoroughly dried, and triturated in the mortar with four to five parts of dry neutral oxalate of potassa and some charcoal dust, or with a mixture of cyanide of potassium and soda, and then heated to redness in a not too narrow glass tube, closed at one end, or better still in a narrow-necked matrass (see Fig .66).

Sulphide of potassium and metallic arsenic are produced, and the latter forms a sublimate, which may be recognized by its lustre and crystalline character, or if only in trifling quantity, may be volatilized and detected as above.

It is very essential in such reduction assays that the mixture to be treated shall be as free from water as possible, and therefore both the substance and the reducing agent should be thoroughly dried.

According to Vogel, the minutest quantities of arsenic, which by the usual method on coal produce no garlic odour, can be detected by mixing the arsenical substance with charcoal dust and a very dilute solution of shellac, and forming from this paste little rolls, which are treated like cracking coals. The glowing coal emits the same garlic odour.

SULPHIDE OF ARSENIC.

Realgar and *orpiment* treated in the matrass fuse, boil, and are sublimed; the former yields a sublimate red when cold; the latter a dark yellow, and both sublimates are transparent. Gently heated in the open tube, they burn and yield sulphurous and arsenous acids. Too strongly heated, a part of the assay is liable to sublime unchanged. On coal they burn with a whitish-yellow flame and grayish-white fumes.

Metallic arsenic may be obtained from sulphide of arsenic in two ways. According to Berzelius, the sulphide carefully heated in the open tube is decomposed into arsenous and sulphurous acids, the former collecting as a crystalline sublimate. The tube should be held inclined and heated a little above the assay, so that the ascending fumes may pass this hottest spot and be perfectly burned. The tube is then drawn out just above the sublimate with the aid of the spirit-lamp, and the arsenous acid then driven into the drawn-out portion and there reduced by an inserted splinter of charcoal, as will be described in detail on a following page.

Another very simple method consists in mixing the sulphide of arsenic, or the sulphur to be tested for arsenic, in the mortar with four volumes of neutral oxalate of potassa and a little charcoal powder, or with six volumes of the mixture of cyanide of potassium and soda, and gradually heating the mixture to fusion in the glass tube or matrass, as before described for sulphide of arsenic.

When the amount of arsenic appears very trifling in comparison with the sulphur, it is as well to remove the excess of the latter by subliming it at a gentle heat in a matrass, and then to break up the matrass and treat the pulverized residue by reduction in a narrow-necked matrass.

SULPHO-ARSENIDES.

The natural sulpho-arsenides yield in the closed tube, according to their varying composition, partly a little sulphide of arsenic with much metallic arsenic, partly sulphide of arsenic alone, while part yield no sublimate whatever. In the open tube they all yield arsenous and sulphurous acids. The metallurgical products containing compound sulphides and arsenides in not too small quantity show the same behaviour.

On coal in R. F. they frequently emit a distinct arsenical odour, but a small quantity of arsenic cannot always be thus perceived, since it either volatilizes in combination with arsenic, or in the presence of cobalt or nickel is not volatilized at all. Such substances, including chiefly many metallurgical products; viz. Rhostein, lead matt, cadmia, etc., may be powdered, mixed with three to four parts of neutral oxalate of potassa, or cyanide of potassium, and fused on coal in R. F. Sulphur is thus separated as sulphide of potassium, and the arsenic, if not combined with cobalt or nickel, volatilizes with its peculiar odour.

When this method yields no satisfactory result, a certain one may be obtained by the process for metallic compounds containing little arsenic, p. 319.

ARSENOLITE (ARSENOUS ACID).

In the matrass sublimes very readily, and the crystalline sublimate often shows distinct octahedra under the magnifier. According to Berzelius, very trifling quantities of arsenous acid may be reduced to metal by using a glass tube drawn out to the diameter of a coarse knitting-needle and sealed at the narrow end. The arsenous acid, which may amount to less than one milligr., is placed in the drawn-out part at *a*, Fig. 67, and over it is inserted a splinter of charcoal, reaching from *c* to *b*. The narrow part, *ab*, is then heated in the spirit-lamp, until the charcoal glows, when the end containing the acid is likewise drawn into the flame. The vapour of arsenous acid passing over the glowing coal is reduced and a metallic coat of arsenic forms in the colder part at *d*. Should the amount of arsenous acid be very trifling indeed, only a black film of arsenic will be produced between *c* and *d*, but by gradually bringing the flame nearer and nearer to this it may be driven together to a ring, and if

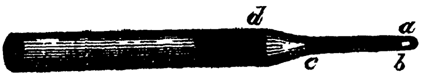

Fig. 67.

the tube is cut off at *c* and the portion *d* held in the flame, the arsenic will be volatilized and afford its characteristic odour.

Heated alone on coal, arsenous acid volatilizes without diffusing any odour, but when mixed with moist oxalate of potassa and treated in R. F. it is reduced to metallic arsenic, which volatilizes and affords the arsenic odour.

Oxide of antimony (antimonous acid), destined for medicinal and pharmaceutical purposes, must be tested for arsenous acid, and if not much less than a thousandth part is present, it will afford a distinct arsenical odour, when the oxide of antimony is treated on coal in R. F. with neutral oxalate of potassa or cyanide of potassium. Should this test give no decisive result, a reduction assay must be made in the matrass, with oxalate of potassa, or a mixture of cyanide of potassium and soda. Oxide of antimony containing less than a thousandth part of arsenous acid, when heated to redness in a narrow-necked matrass with three volumes of the neutral oxalate and one of charcoal dust, will afford a very distinct metallic mirror, which on further treatment in the spirit flame is volatilized with an unmistakable arsenic odour.

ARSENIC ACID.

Strongly ignited in the matrass it is converted into arsenous acid, which sublimes, and oxygen which escapes. On coal it is reduced to metal, which volatilizes immediately and diffuses a strong arsenic odour.

COMPOUNDS OF THE ACIDS OF ARSENIC WITH EARTHS AND METALLIC OXIDES.

These compounds may be tested for arsenic in various ways. A few arsenates can be recognized by the crystalline arsenous acid which they yield in the matrass (*vide* arsenates of iron, cobalt, and nickel). The greater number are, however, recognized by the light-blue colour which they impart to the flame when tested in the forceps, provided the bases themselves do not colour the flame intensely. When the presence of arsenic or arsenous acids cannot be detected by testing the salts alone, proper reagents must be employed.

A very simple method of recognizing arsenates consists in mixing the powder with soda, or better still, with neutral oxalate of potassa, or cyanide of potassium, and treating it in R. F. on coal, when the odour will show whether metallic arsenic is liberated or not. This test is not, however, sufficiently decisive in all cases, especially when the acids of arsenic are combined in trifling quantity with metallic oxides, which are easily reduced and then form fusible compounds with the arsenic, from which the latter can only be separated with difficulty. Unless much arsenic were present it might happen that none of it would then be liberated. In such cases the process described for detecting small quantities of arsenic in metallic compounds, p. 319, is followed, but in place of the nitre a mixture of equal parts of nitre and soda is used for the fusion.

Arsenates and arsenites of bases which are reduced with difficulty, or when easily reducible have no great affinity for arsenic, can be tested by mixing the fine powder with three to four volumes of neutral oxalate of potassa, or a mixture of cyanide of potassium and soda, and fusing it in a matrass. Regard must be had to all that has been said on p. 319. Quite distinct mirrors are obtained from salts of the earths, or oxide of silver; somewhat less distinct mirrors from salts of iron or copper.

25. TELLURIUM, Te.

Its occurrence in the mineral kingdom.

Tellurium usually occurs in the *metallic* state:

a. Alone in

Native tellurium, Te, which is seldom free from other metals, as gold and iron.

b. Combined with other *metals:*

Melonite;

Altaite and nagyagite (*vide* lead);

Tetradymite (*vide* bismuth);

Hessite and petzite (*vide* silver);

Sylvanite (*müllerite*) (*vide* gold).

Tellurite (*tellurous acid*),—Te, is said to accompany native tellurium occasionally.

Montanite (*vide* bismuth).

Examination for Tellurium.

Native tellurium in the open tube fuses, burns with a bluish-green flame, and emits fumes. The fumes collect within the tube to a grayish-white sublimate, which when strongly heated is converted entirely into tellurous acid and fuses to clear, transparent drops. On coal it behaves as stated on p. 65, but the volatilizing tellurium generally leaves a slight residue, which treated with borax and a little test lead in R. F. imparts an iron colour to the glass, and the lead yields by cupellation a small gold button.

Compound substances may be tested for tellurium both in the open tube and on coal. The behaviour of the above-named compounds of tellurium with other metals in the open tube is given in the corresponding places, but it may be generally remarked that in roasting a mineral containing tellurium in the open tube, the latter volatilizes more or less completely, is changed to tellurous acid and forms white fumes, which condense rather near the assay. On heating the tube where the coat is thickest with the blowpipe flame, the acid fuses to clear, colourless drops, most distinctly perceived with the magnifier. It should not be heated too strongly or too long, however, as tellurous acid does not withstand the heat under access of air. Should much lead be present, a gray sublimate forms near the assay and a white one further from it. The latter fuses to colourless drops with a moderate heat, and therefore consists of tellurous acid, while the gray sublimate does not fuse to drops, but is altered to a half-fused, grayish film on the glass. According to Berzelius, it is tellurate of lead.

When bismuth is present, it remains behind, and the tellurium sublimes as tellurous acid. On continued heating, the remaining metal oxidizes on the surface, and is surrounded by fusing, brown oxide of bismuth.

In many cases the examination for tellurium may also be advantageously conducted in the closed tube. known that tellurium in combination with potassium or sodium forms a purple solution in boiling-hot water, and thus its presence can be detected. According to Berzelius, it is therefore only necessary to triturate the substance with soda and some charcoal, fuse the mixture in the closed tube, and after it is cold to drop a little boiling water into the tube. After a time this assumes a more or less intense purple colour, from the dissolved telluride of sodium.

This test is applicable not only to substances containing metallic tellurium, but also to the acids of tellurium, which are thus reduced.

According to von Kobell, the natural tellurium compounds when gently heated in a matrass with much concentrated sulphuric acid, impart to it a purple or hyacinth-red colour, which disappears on adding water, while a blackish-gray precipitate is formed. When a mineral containing tellurium is treated on coal, it generally yields a white tellurous acid coat, with a reddish-yellow border, which disappears under the R. F., imparting to the flame a green, or in presence of selenium a bluish-green, tinge. Should the horseradish odour be also perceived, this is a certain indication of selenium.

If the mineral contains lead or bismuth and is treated alone on coal for only a few moments, no pure tellurous acid coat is obtained, but a mixture of this with oxide of lead or bismuth is liable to be deposited. This difficulty can be remedied by mixing the powdered assay with an equal volume of vitrified boracic acid and treating it in R. F. The oxide of lead or bismuth is dissolved in the boracic acid, notwithstanding the reducing flame, and yields no coat, while the tellurium alone volatilizes and coats the coal. When much selenium is also present, a portion of it is deposited on the coal, and then the tellurous acid coat is less distinct. In such cases the mineral must also be tested in the open tube.

Melonite in the open tube gives a sublimate fusible to colourless drops, and leaves a gray mass; on coal burns with a bluish flame, giving a white, volatile coating and a greenish-gray residue; in R. F. with soda a gray powder of magnetic, metallic nickel. (Dana.)

C. Examination for non-Metallic Elements and Acids.

1. OXYGEN, O, AND HYDROGEN, H, IN COMBINATION AS WATER,—H.

Occurrence of water in the mineral kingdom.

It forms an essential constituent of most natural salts, many silicates, and the natural hydrates, but also occurs only as an accidental ingredient of many minerals, as shown by their respective chemical composition, already given in various places.

Examination for Water.

This is performed very simply in a matrass which has been freed from moist air by warming and drying it, after which it is gradually heated in the spirit flame. If the substance contains mechanically combined water, or is a salt which contains chemically combined water and is itself soluble in water, the combined water will, in the former case be entirely vaporized by the first action of the heat, and in the latter case partly The vapour condenses in the narrow, cold part of the neck t drops, which are plainly visible. A substance which is no soluble in water seldom yields its chemically combined wate at first, but when the matrass is heated to redness, the wate escapes and condenses as before. To obtain a distinct wate reaction from silicates containing little water, they must b powdered, and, if necessary, heated with the blowpipe. Som phenomena which may occur while testing for water hav been especially noticed on pp. 57–9.

2. NITROGEN, N, AND OXYGEN, O, COMBINED AS NITRIC ACID,—N.

This acid occurs with potassa in *nitre* (*vide* potassa); wit soda in *soda nitre* (*vide* soda); and with lime in *nitrocalcit* (*vide* lime)

Examination for Nitric Acid,

Including the blowpipe characteristics of nitrates in general.

Nitrates, part of which fuse in the matrass when heated, are thereby decomposed more or less readily. When the acid is combined with strong bases, oxygen alone is at first liberated, but in so small quantities that it cannot be recognized by means of a glowing splinter; nitrites remain, which are perfectly decomposed only by a very strong heat. Nitrate of ammonia fuses very easily and is decomposed, with ebullition, into water, and protoxide of nitrogen; if heated too quickly or strongly, or in a matrass with a narrow neck, an explosion is liable to occur. Salts with weaker bases evolve at a moderate heat oxygen and nitrous acid, the latter being recognized by its yellow colour and its odour.

When nitrates of the fixed alkalies, or alkaline earths, are so strongly heated on coal, that the coal in contact with them glows, they deflagrate violently and are converted into carbonates. Other nitrates deflagrate less vividly, and leave their bases as earths, metallic oxides, or, in case the latter, are easily reduced, as metals, which if volatile pass off partly or entirely in fumes, and coat the coal.

A small amount of a nitrate present in another salt or substance can be readily detected by heating it with rather more than its volume of bisulphate of potassa in the closed tube or matrass. The tube is then filled with gaseous nitrous acid, the yellow colour of which may be most clearly seen by looking down through the tube. Should there be so little nitrate present that this colour cannot be plainly seen, the minutest quantities may, according to Stein, be detected by heating the assay with litharge, which at first absorbs the nitric acid, but yields it up at a higher temperature.

A slip of filter-paper which has been immersed in a solution of protosulphate of iron, free from sesquioxide and acidulated with some sulphuric acid, is inserted into the neck of the tube, and, if nitrous acid is present, will assume a yellowish to brown colour. In this way the nitric acid in a mixture of 1 part of nitre with 1000 parts of sulphate of soda, containing only 0·0005 nitric acid, can be distincly shown. The paper quickly loses its colour if too strongly heated, and therefore the tube or matrass should be rather long.* *Nitre, soda nitre,* and *nitrocalcide* are immediately recognized as nitrates by the above tests, and their bases may be distinguished by the colour they impart to the flame.

3. CARBON, C, AND CARBONIC ACID, C.

Occurrence of carbon and carbonic acid in the mineral kingdom and in metallurgical products.

Carbon occurs in nature

a. Alone in

Diamond;
Anthracite;
Graphite;
Black-band from Westphalia and England;
Pyrorthite (*vide* cerium);
Slate (*vide* potassa).

b. Combined with *hydrogen;*

c. Combined with *hydrogen* and *oxygen;*

d. Combined with *oxygen* as *carbonic acid.*

Carbonic acid is found in nature, both as a free gas, and combined with bases in many minerals, which have already been enumerated.

Small quantities of carbon are likewise disseminated throughout several minerals, either free, or combined with hydrogen, oxygen, or nitrogen. In metallurgical products carbon forms an essential constituent of *raw iron* and *steel,* while other products, especially iron *bears,* frequently contain a little carbon (*vide* iron).

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* If the litharge contains peroxide of lead and there are metallic chlorides present, hydrochloric acid or chlorine is evolved by the heat, and these also colour the paper. The presence of the peroxide is detected by heating the litharge with salt and bisulphate of potassa, when chlorine is liberated, and can be recognized by its odour or by its bleaching effect on indigo-paper.

Examination for Carbon and Carbonic Acid,

Including the blowpipe characteristics of the minerals belonging here.

Diamond.—The experiments of Petzholdt indicate that no extraordinarily high temperature is required for the combustion of diamond, but that diamond dust, produced by rubbing two diamonds together, may be burned on platinum foil over the spirit-lamp, and it is said to glow as brightly as coal dust under similar treatment. A small diamond placed on the foil is said to be likewise entirely consumed by directing the blowpipe flame down upon it, while toward the end it glows brightly. A very high temperature is necessary only in burning larger diamonds. The product of combustion is carbonic acid gas.

Anthracite usually yields in the matrass moisture, but no empyreumatic oil; it is not combustible in the candle-flame. Heated in the platinum spoon with the O. F. it burns very slowly, without flame, and leaves an ash, containing more or less iron.

Graphite sometimes yields considerable water. Heated over the spirit-lamp in the platinum spoon it is unaltered; in the forceps in O. F. it gradually decreases in volume. The streak produced by it on fire-clay and ignited in the O. F., until all the carbon is gone, frequently becomes red from oxide of iron. The powder heated to redness with nitre in the platinum spoon deflagrates, and after washing away the salt, now chiefly altered to carbonate of potassa, earthy and metallic admixtures remain, which may be further tested as for silicates (*see* lime).

Black band from Westphalia yields water, and a feebly bituminous odour in the matrass. B. B. on coal, burns reddish-brown. Acids liberate carbonic acid; when boiled with aqua regia, it finally leaves only coal and a trace of silica.

Inflammable cinnabar (*Quecksilberbranderz*) from Idria, containing *idrialite,* fuses in the matrass and evolves mercurial and sulphurous vapours and heavy carburetted hydrogen, leaving a coaly, porous residue. B. B. on a clay capsule it takes fire and burns, evolving smoke and sulphurous acid, and leaving a brownish-red ash.

Kœnlite fuses at 114° C., and is decomposed with ebullition at 200°. It leaves a coaly residue.

Ozocerite fuses in the candle-flame to a clear, oily fluid, which solidifies on cooling. At a higher heat it burns with a flame and volatilizes, sometimes leaving a slight coaly residue.

Scheererite, according to Stromeyer, fuses at 45° C. to a colourless fluid, which on cooling forms a radiated mass. Above 100° it volatilizes and condenses in acicular crystals. It burns completely with a somewhat sooty flame and slight odour.

Asphaltum fuses very easily in the matrass, evolving an empyreumatic oil, a little ammoniacal water, and combustible gases, and leaving a coaly residue, which yields by combustion on a clay capsule an ash, consisting chiefly of silica, alumina, and sesquioxide of iron. It burns with a bright flame and much smoke.

Retinite from Halle fuses in the matrass, blackens, and yields a brown, thick oil and acid water. It burns with a bright flame and much smoke.

Amber fuses with some difficulty in the matrass, yields water, empyreumatic oil, succinic acid, and gases, and leaves amber resin (*Bernsteincolophonium*).

It burns with a bright flame and a peculiar agreeable odour.

Bituminous coal heated in the matrass is infusible (close-burning coal); or *sinters* together (open-burning coal); or becomes soft and swells up (caking coal). It evolves in all cases empyreumatic products and combustible gases, often including sulphuretted hydrogen. The residue is coke, having a more or less metallic lustre, which takes fire with difficulty in the air and behaves like anthracite.

Held in the candle-flame, or heated B. B. on the clay capsule, it burns with a luminous, smoky flame, and when all the coal is consumed leaves an ash, consisting of silica, alumina, lime (gypsum), and sesquioxide of iron.

Brown coal is infusible in the matrass, but some varieties are somewhat softened; on further heating they evolve combustible gases, acid water, empyreumatic oils, and a peculiar, disagreeable odour. There is a large residue, which tested in the open air behaves like coal, and often leaves a considerable ash.

It burns in the candle-flame, or on the clay capsule, with a smoky flame and disagreeable odour.

According to von Kobell, *bituminous coals* and *asphaltum* boiled with potassa lye, impart to it only a feeble yellowish colour, or none at all. If their powder is boiled with ether in a matrass, or closed tube, which is placed in hot water, asphaltum colours it wine- or brownish-red, but the coals only pale yellowish, or not at all. Asphaltum is also much more fusible than the most fusible bituminous coals, and melts in drops in the candle-flame.

The *brown coals* are readily distinguished by the brown colour which they impart to boiling potassa lye. The bituminous coals ignited in the candle-flame, or before the blowpipe, are immediately extinguished when removed from the flame, but the brown coals continue to glow for some time.

Dysodile, from Gleimbach near Giessen, burns with a flame and a disagreeable odour. In the matrass it yields water and a yellow empyreumatic liquid. B. B. it scales off, and after the organic portions are destroyed leaves a red residue, which strongly heated fuses to a reddish-brown slag, that scratches glass and is not attacked by acids, but yields silica with potassa. With borax and S. Ph. it shows iron and silica.

In minerals and other substances, excepting metals and their combinations, which contain carbon, or compounds of carbon and hydrogen, with a trifling admixture of oxygen or nitrogen, the carbon can be detected by ignition with antimonate of potassa (p. 48). The fine powder is mixed in the agate mortar with two to three volumes of the antimonate, if consisting chiefly of earthy matters, and with six to eight volumes if containing metallic sulphides, and then heated to redness in a matrass over the spirit-lamp. The carbon is oxidized at the expense of the antimonic acid, forming carbonic acid, which combines with the liberated potassa, while if a notable amount of carbon or sulphur is present, a little oxide of antimony volatilizes and partly condenses in the neck. When the substance consists of metallic sulphides, sulphate of potassa and a little sulphide of potassium are formed. When cold, the matrass is filled nearly to the neck with water, which is gradually heated to boiling. The carbonate and sulphate of potassa dissolve, with part of the undecomposed antimonate of potassa, while most of the latter remains with the earths and metallic oxides.

To the quite warm solution a few drops of nitric acid are added, which causes effervescence, more or less lively according to the amount of carbon present. Not a bubble will be seen to ascend if the substance contained no carbon, but several will be perceived if a trifling amount of carbon was present. It must be observed, however, that the solution should be warm enough to allow the carbonic acid to escape.

Carbonates vary as to their behaviour in the matrass. When combined with earths or metallic oxides, the carbonic acid frequently escapes below a red heat, while many of the oxides become more highly oxidized, like protoxide of iron. Carbonate of magnesia is completely decomposed at a red heat; carbonate of lime only imperfectly, but it can be entirely decomposed by moistening the undecomposed salt with water and again strongly igniting it. Carbonates of the fixed alkalies, strontia and baryta, alone are not altered , carbonate of ammonia sublimes unchanged.

All the non-volatile carbonates are decomposed on coal; the carbonates of the alkalies and baryta fuse, sink into the coal, evolve carbonic oxide, and react very strongly alkaline on moistened litmus-paper. Carbonates of the alkalies fuse with silica on platinum wire, or on coal, with effervescence to a clear, colourless glass; they also yield their carbonic acid with effervescence when fused with borax or S. Ph., as do also the other carbonates.

The simplest method of detecting carbonic acid with certainty in any substance consists in pouring a little dilute nitric acid upon it in a glass vessel and observing whether any effervescence ensues; in case sulphides are certainly absent, dilute hydrochloric acid may be used. The glass should be slightly heated if no gas is otherwise evolved. It is not well to use concentrated acids, since many carbonates are only soluble in dilute acids, *e. g.* witherite,

Examination for Carbon in Metallurgical Products.

In *raw iron, steel,* and *bears,* the carbon, whether chemically combined, or only disseminated as graphite, is most simply found by digesting a small fragment in a porcelain dish, with about six times its weight of fused chloride of silver and some water acidulated with a few drops of hydrochloric acid, leaving the whole covered with a watch-glass, until all the iron is dissolved. The iron is converted into protochloride, the carbon remains behind, and a corresponding amount of silver is reduced. The carbon generally contains earthy matters, and may, if desired, be tested with antimonate of potassa, p. 332.

Metallurgical products, consisting chiefly of metallic sulphides, may also be tested for disseminated coal with antimonate of potassa.

4. BORON, B, AND BORACIC ACID, B.

Occurrence of boracic acid in the mineral kingdom.

It occurs combined with

a. Water in

Sassolite.

b. With *soda* in

Borax (*vide* soda).

c. With *ammonia* in

Larderellite (*vide* ammonia).

d. With *earths* and *metallic oxides* in

Boracite (*vide* magnesia);
Ulexide,
Borocalcite,
Hydroborocalcite,
Hydroboracite,
vide lime;
Sussexite (*vide* manganese).

e. In *silicates,* especially in

Datolite,
Botryolite,
Axinite,
Danburite,
vide lime;
Tourmaline (*vide* magnesia).

Examination for Boracic Acid,

Including the blowpipe characteristics of the minerals containing it.

Sassolite yields in the matrass water and a little ammonia.

On platinum wire or coal fuses, with intumescence, to a clear glass, and tinges the flame yellowish-green. If gypsum is present, the glass becomes cloudy on cooling.

Borax in the matrass yields much water and puffs up, becoming black in the hottest parts, from charring of the adherent organic matters; a burnt odour is also perceptible.

B. B. fuses with intumescence to a clear, colourless bead, and yields a soda flame. The reaction of boracic acid is only obtained by the special test, soon to be described.

Larderellite yields in the matrass water and ammonia and a slight white sublimate; at a higher heat it fuses. On platinum wire shows pure boracic acid reactions.

All the borates intumesce more or less when heated, and then fuse to a bead. The bases, if volatile, are removed, *e. g.*, ammonia and oxide of mercury, and leave pure boracic acid. When the bases do not colour the flame, the salt affords the yellowish-green boracic acid flame. When no such flame is produced, or when the substance containing the boracic acid does not also colour the flame green, the acid may frequently be detected by using sulphuric acid.

Turner has proposed a test for boracic acid in salts and minerals as follows :—The fine powder is mixed to a paste with a little water and one part of a flux, consisting of four and one-half parts bisulphate of potassa and one part finely-powdered fluorspar, perfectly free from boracic acid. It is then fused on platinum wire within the blue flame, and as soon as the water is expelled, fluoboracic acid is formed, which is volatilized and imparts a yellowish-green tinge to the flame. This coloration is very transient, however, and must be looked for with great attention, if little boracic acid is present. According to Merlet, three to four parts of the flux are requisite to obtain a sure result.

5. SILICIUM, Si, AND SILICIC ACID, $\dot{\mathrm{S}}\mathrm{i}$.

The occurrence of silicic acid in the mineral kingdom and in metallurgical products.

Silicic acid occurs very frequently in nature, partly free, partly combined with water, and partly with various bases, with which it forms natural silicates.

a. In the free state it constitutes *quartz*, of which several varieties are distinguished:

Rock crystal, smoky quartz;
Amethyst;
Ordinary quartz.

Among the varieties, distinguished by colour, lustre, or structure are:—Rose quartz, milky quartz, siderite or sapphire-quartz (of indigo or Berlin-blue colour), prase (leek-green), cat's eye (greenish-white to greenish-gray and olive-green, also red and brown), aventurine (yellow, red, or brown, with numerous minute scales of mica, or small fissures in different directions), fibrous quartz, in parallel fibres, aggregated into flattened groups.

Ferruginous quartz;
Hornstone;
Lydian stone (*touchstone*);
Jasper;
Chalcedony, carnelian, onyx, sardonyx, heliotrope, plasma, Mochastone;
Chrysoprase;
Flint;
Agate, composed of several varieties of quartz intermixed; viz. amethyst, chalcedony, and jasper.

b. Combined with *water* silicic acid forms

Opal, essentially amorphous silicic acid with 0·1 to 13 per cent. water;

It includes

Precious opal, fire-opal, common opal, hydrophane, semiopal, hyalite, menilite, cacholong, jasp-opal (*Eisen-Opal*) and fiorite (siliceous or pearl sinter).

Finally are to be here included:

Float-stone and alumocalcite, containing alumina and lime,
Tripoli slate, tripolite, and infusorial earth.

c. In combination with various bases silicic acid forms a great number of *natural silicates,* which have been already enumerated. It is also a chief constituent of many ores, dressed on the large scale, and of most slags, while minute quantities of silicium also occur in *raw iron, steel,* and certain *bears* (see iron).

Examination for Silicic Acid,

Including the general blowpipe characteristics of the above-named minerals and metallurgical products.

The minerals enumerated under *quartz, a,* yield in the matrass no water, or only traces. B. B. they are quite infusible. Their powder dissolves slowly in borax to a clear, difficultly fusible glass, which while hot is frequently coloured by the metallic oxides present. They are scarcely attaked by S. Ph.; with soda they fuse with effervescence to a clear glass.

The minerals enumerated under *opal, b,* yield more or less water in the matrass and lose their lustre. In the forceps they are infusible, and if quickly heated decrepitate. With the fluxes they behave like the preceding.

The *silicates,* both natural and artificial, can be recognized by means of S. Ph. and soda. They are nearly all decomposed by S. Ph., the bases combining with the free phosphoric acid and leaving the silica undissolved. The test is performed on platinum wire by first fusing the S. Ph. to a bead and then attaching to this, while soft, a few very fine splinters of the silicate and treating it for a sufficient time in O. F. If the silicate can be thus decomposed, the bases dissolve, leaving a silica skeleton, which floats in the hot, clear bead. If the bases yield with S. Ph. at a certain saturation, either by flaming, or on cooling, a milk-white or opalescent bead, *e. g.,* lime, magnesia, glucina, or yttria, the bead will be more or less cloudy on cooling, and the presence of separated silica must be ascertained while it is hot. Should the silicate not be decomposed thus, it must be tested in powder, when, if decomposable, it will leave a gelatinous residue of silica. Silicates of which the bases are chiefly zirconia, cannot be perfectly decomposed, even in fine powder, and their silica is best found as described under zirconia.

When a substance contains but little of a silicate, or only some disseminated quartz, the silica will be dissolved and the S. Ph. bead shows no signs of separated silicic acid; but it may be detected in the wet way, by proceeding as directed for silicates under lime, magnesia, and alumina. The silica when thus separated can be easily recognized by means of S. Ph. or soda.

With soda on coal and on platinum wire the silicates dissolve with effervescence, some perfectly, some only partially. For special descriptions, *vide* p. 90.

When a compound of oxidized substances, which are not reducible by soda, contains a sufficient amount of a silicate, a slight effervescence may be observed when testing it with soda, from which the presence of silicic acid may be inferred, provided the substance is free from other acids that withstand the fire. It is, however, always safer to employ the wet way.

The silicium in *raw iron*, *steel*, and *bears* may be found by dissolving the product in nitric acid, or decomposing it with chloride of silver (p. 334), when silica, carbon, etc., remain behind. The residue is collected on a filter, incinerated in the platinum spoon, and the remaining silica tested with soda on coal.

6. Sulphur, S, and Sulphuric Acid, S.

Their occurrence in the mineral kingdom and in metallurgical products.

Sulphur occurs in nature:

a. As native Sulphur, frequently rendered impure by quartz, lime, iron, coal, water, etc.

b. Combined with many *metals*.

Sulphuric acid occurs in combination with alkalies, earths, and metallic oxides.

All the minerals containing sulphur or sulphuric acid have been mentioned.

Among metallurgical products sulphur forms a chief ingredient of the *matts* (Stein and Leche), which have already been enumerated under the respective metals, and is occasionally an accessory ingredient in certain raw metals and compounds of metals, which are to be submittêd to further treatment, as in raw iron; also in certain slags.

Sulphuric acid constitutes a chief ingredient of artificial sulphates (vitriols), and occurs in greater or less quantity, combined with earths and metallic oxides, in ores roasted on the large scale, which are to be treated for their metals, or the production of alum, or sulphate of iron, zinc, or copper.

Examination for Sulphur and Sulphuric Acid,

Including the general blowpipe characteristics of sulphates and sulphites.

Native sulphur fuses very easily in the matrass and sublimes with a brownish colour, but becomes yellow again on cooling; foreign ingredients, if non-volatile, remain behind. Ignited on coal it burns with a bluish flame, evolving sulphurous acid, which is recognized by its characteristic pungent odour.

In its combination with metals sulphur can be detected in various ways:

a. In certain cases by heating the substance strongly in the closed tube. Some sulphides containing a high proportion of sulphur yield a sublimate of the latter, *e. g.*, $\overset{\prime\prime}{\mathrm{Fe}}$, $\overset{\prime\prime\prime}{\mathrm{Fe}}$, $\overset{\prime\prime}{\mathrm{Mn}}$, $\overset{\prime}{\mathrm{Cu}}$. If the sulphur is combined with volatile metals, as *mercury* or *arsenic*, it sublimes in combination with the metal, and the sublimate may be recognized by its colour (*vide* sulphide of arsenic, pp. 60, 321, and cinnabar, pp. 60, 276). When the sulphur is combined with antimony, a strong heat produces the sublimate of $\overset{\prime\prime\prime}{\mathrm{Sb}}$ with Sb, already mentioned.

b. By roasting in the open tube. Although a trifling amount of sulphur may not always produce the odour of sulphurous acid, yet the latter will redden an inserted strip of moistened blue litmus-paper. Substances which amount of metallic sulphides and yield no sulphurous acid when in fragments, will do so if treated in the powdered state.

c. By heating the substance on coal in O. F. If there is but little sulphur, however, the odour of sulphurous acid will not always be perceptible.

d. In most cases even a very little sulphur may be detected by fusing the powdered substance with two parts of soda, perfectly free from sulphate, and one part borax on coal in R. F., provided no selenium is present. In the case of easily-fusible metals, which contain only finely-disseminated sulphides and cannot be pulverized, *e. g.*, raw lead, black copper, etc., a fragment the size of a mustard-seed or small pepper-corn is used; in case of metals that fuse with difficulty, as raw iron, the necessary amount must be obtained by filing. While the powdered substance is fused with the soda and borax in R. F., or the glass treated by the side of the metal for some time, sulphide of sodium forms, which immediately yields a sulphur reaction when the fused mass is removed from the coal, pulverized, placed on a bright sheet of silver and moistened with water. Sulphuretted hydrogen is evolved, which colours the silver quite black, with sulphide of silver, if a notable amount of sulphur is present; but if less is present, only dark-brown or yellow. The resulting stain may be readily removed by rubbing it with moistened charcoal or fine bone-ash. The borax acts advantageously by preventing the sulphide of sodium from sinking into the coal and by forming with it a mass which is readily removed.

Since selenium forms selenide of sodium, which has a similar effect on silver, the substance must always be previously tested alone on coal, to ascertain whether a selenium odour is perceptible. Should selenium and sulphur occur together, the test for sulphur must be performed in the open tube and the formation of sulphurous acid ascertained by the odour, or with moistened litmus-paper.

Sulphates.

The *sulphates* behave variously in the matrass and on coal. The sulphates of *alkalies, alkaline earths,* and *lead,* are not at all decomposed in the matrass. An imperfect decomposition ensues with the salts of other strong bases, viz. *protoxides of iron and manganese* and *oxide of zinc;*

the heat requisite for their complete decomposition cannot be produced. The salts of *non-alkaline earths* and the *weaker metallic bases* are more or less readily decomposed. When the salt suffers partial decomposition, sulphurous acid is evolved and may be recognized as usual.

On coal, especially in R. F., the sulphates of the fixed *alkalies* and *alkaline earths* are converted into sulphides, with an alkaline reaction, and the former, after sinking into the coal, are partially volatilized and form a white coat. If moistened with water, or, better still, dilute hydrochloric acid, these sulphides evolve sulphuretted hydrogen. The remaining sulphates evolve sulphurous acid and leave partly earths or metallic oxides, partly reguline metals, or metals containing sulphur, provided the reduced metal is not volatile; if it is, a coat of oxide is deposited.

Sulphites.

The *sulphites* are all decomposed in the matrass, leaving either pure oxide, or a mixtnre of basic sulphates with sulphides, so that after ignition the sulphites of the alkalies or alkaline earths evolve sulphuretted hydrogen, if moistened with dilute hydrochloric acid. On coal they behave like the sulphates.

There are two ways of dctecting sulphuric or sulphurous acids in sulphates or sulphites and in other salts containing small quantities of these acids.

a. When the bases of the salts produce no coloration in the glass fluxes, sulphuric acid can be detected by forming with soda and silica on coal in R. F. a bead, which is perfectly clear and colourless, and then fusing this bead with a little of the salt in R. F. and observing the colour of the cold glass. The sulphuric acid is reduced, forming sulphide of sodium, which produces a yellow to dark-red colour, according to the amount of sulphuric acid present. Should tho bases be metallic oxides which colour the fluxes, the salt must first be decomposed by mixing it with once or twice its amount of soda, igniting the mixture in O. F. on platinum foil or wire, dissolving the resulting sulphate of soda in a few drops of water, evaporating the clear solution to dryness on platinum foil, or in a porcelain dish, and testing the salt as above with silicate of soda.

b. The test for sulphuric acid may be performed by mixing the salt to be tested with soda, or, if this not quite free from sulphate of soda, with neutral oxalate of potassa, fusing it in R. F. on coal, removing the fused mass from the coal, into which part of it will have sunk, and moistening it on silver foil. The fused mass may also be covered with dilute hydrochloric acid in a matrass, and the presence of sulphuretted hydrogen tested by inserting a strip of filter-paper moistened with solution of acetate of lead into the mouth of the matrass. If sulphuretted hydrogen is evolved, it will form sulphide of lead, and colour the paper black or brown.

Dana has proposed the following test for sulphur. The substance is fused with soda in R. F., the assay moistened with a drop of water in a watch-glass and a bit of nitroprusside of sodium, as large as a pinhead, added. If sulphur in any form was present, a purple colour will be produced, to which Playfair first called attention. It is well to add some starch to the soda when using this test for organic substances; viz., horn, hair, nails, etc. A bit of hair four inches long, wound upon platinum wire and dipped in the mixture of starch and soda, is said to yield an unmistakable sulphur reaction.

Molybdate of ammonia is, according to Schlossberger, an exceedingly sensitive reagent for sulphur. A dilute solution of it supersaturated with hydrochloric acid, is coloured fine blue by very small quantities of sulphuretted hydrogen or sulphides dissolved in water.

In order to determine whether the sulphur in a mineral is present as a sulphide or sulphate, von Kobell recommends that the powdered substance should be fused with potassa in the platinum spoon before the blowpipe. The spoon with its contents is then placed, with a strip of sheet silver, in a small porcelain vessel and covered with water. The mass dissolves, and after some time the silver will either become black, or remain bright. In the former case the substance contains a sulphide, *e. g.*, haüynite, helvite, etc.; in the latter case a sulphate, provided the presence of sulphur has been already ascertained by the test with soda on coal. Naturally the substance must be perfectly free from any ingredient which could have a reducing action.

7. SELENIUM, Se.

Its occurrence in the mineral kingdom.

Selenium occurs only in combination with metals: in clausthalite, tilkerodite, zorgite, and lehrbachite (*vide* lead); berzelianite (*vide* copper); tiemannite (*vide* mercury); naumannite, crookesite, and eucairite (*vide* silver). It sometimes forms an unessential ingredient of tellurium minerals, and certain galenas, pyrites, and phosphates of copper.

Examination for Selenium.

The test for selenium is so simple that even a trace of it can be detected in any substance.

For the behaviour of *tiemannite vide* mercury.

Compounds which are not volatile in the closed tube are tested as follows for selenium: A fragment is ignited on coal in O. F. and immediately held under the nose, when any selenium present will yield the peculiar horseradish odour of the gaseous oxide. If the substance contains much selenium, brown fumes, consisting only of finely-divided selenium, will be evident, before the assay begins to glow; afterward a steel-gray, metallic coat forms, which sometimes has a red border. Selenium may also be separated from its combinations by heating the substance in the open tube, inclining it so that the other constituents may be oxidized; the selenium then separates and condenses in the tube with a red colour. If there is much selenium, the sublimate appears rather steel-gray near the assay. Occasionally also small crystals of selenous acid are deposited beyond the red sublimate, but they volatilize at a gentle heat. If sulphur is likewise present, it escapes as sulphurous acid, and is thus recognized. When a trifling amount of selenium occurs with tellurium, as in *tetradymite*, and the assay is conducted in the open tube, tellurous acid at first collects on the glass, and after continued heating with the blowpipe this appears to be mingled in one spot with a red substance, which consists of selenium.

Selenates and *selenites* are reduced in the R. F. on coal to selenides, which emit a distinct horseradish odour. With addition of soda the reduction is more rapid.

8. PHOSPHORUS, P, AND PHOSPHORIC ACID, $\ddot{\text{P}}$.

Their occurrence in the mineral kingdom and in metallurgical products.

Phosphoric acid is always found in nature in combination with bases, and occurs as an essential constituent in various minerals, which have been enumerated under lithia, lime, magnesia, alumina, yttria, manganese, iron, lead, uranium, and copper.

The slags produced in smelting silver, lead, or copper ores containing phosphates, always hold some phosphoric acid, which may be combined with various bases; iron refining-cinders also frequently contain combinations of phosphoric acid, as the raw iron is not always free from phosphide of iron.

Examination for Phosphorus and Phosphoric Acid,

Including the general blowpipe characteristics of phosphates.

The examination for phosphorus occurs chiefly in case of raw iron. A fragment of the iron weighing about 100 milligr. is dissolved by warming it with nitric acid, which converts the phosphorus into phosphoric acid and leaves the graphite behind. After evaporating to dryness in a porcelain dish, the dry mass is strongly heated, until it evolves no more acid vapours, and is then tested for phosphoric acid (*vide a* to *d* below).

Phosphates. *Phosphates* are not decomposed by ignition in the matrass, but some are fusible. In the forceps, or on platinum wire, most of them can be fused, especially the acid salts, and they colour the flame pale bluish-green, provided the bases produce no coloration. On coal most phosphates can also be fused without decomposition, since the combined phosphoric acid is either not reduced at all, or only very imperfectly. The most evident example of this is neutral phosphate of lead, which fuses very easily to a bead on coal, but scarcely suffers any decomposition in the R. F.: the bead is crystalline (*vide* 244, *et seq.*). Upon fusion with soda on platinum wire, or in the platinum spoon, the phosphates yield phosphate of soda and the bases are liberated.

The test for phosphoric acid may be variously made :—

a. By the pale bluish-green colour imparted to the flame.

b. When the substance contains more than four to five per cent. it may be tested, according to Berzelius, by dissolving some of it in vitrified boracic acid, preferably on coal in O. F., inserting into the fluid glass a bit of fine iron wire, rather longer than the diameter of the bead, and then employing a strong R. F. The iron oxidizes at the expense of the phosphoric acid, producing borate of iron and phosphide of iron, the latter fusing by a strong heat. The glass at first covers the wire, but reassumes its spherical shape when the phosphide fuses, and while the globule is cooling a glowing may generally be observed in the portion attached to the coal, arising from the crystallization of the phosphide of iron. When quite cold, the glass is carefully broken between paper and the spherical metallic button of phosphide of iron subjected to further examination.

It should be magnetic and brittle, and its fracture should show an iron colour. The brittleness of the phosphide depends upon the amount of phosphoric acid that was present. If the substance contained little phosphoric acid, the button may even be somewhat malleable and withstand rather strong blows of the hammer before breaking. When very little or no phosphoric acid was present, the wire will fall from the broken glass bead in its original shape, being only burnt on the ends which projected from the bead.

Should the substance tested for phosphoric acid contain other constituents which can be reduced by the iron and melted to a button with the excess of iron, *e.g.*, sulphuric and arsenic acids, or metallic oxides reducible by iron, their radicals will be obtained in combination with the iron, and a preliminary test for such bodies must be made.

c. Bunsen has proposed a test for trifling quantities of phosphoric acid, which consists in mixing the substance with two to three times as much soda and transferring the thoroughly dried mixture to the drawn-out portion of a tube, somewhat larger than that represented for arsenic. After again heating the mixture so as to remove all moisture, a long bit of sodium is inserted into it and the mixture fused with the blowpipe. When cold, the portion of the tube containing the fused mass is broken off, laid in a porcelain dish and moistened with a few drops of water; if phosphoric acid was present, the well-known odour of phosphuretted hydrogen, resembling decaying fish, will be evolved.

d. The wet way may be employed to detect phosphoric acid in its combinations, and cannot be dispensed with when a substance contains so little phosphoric acid as to produce no reaction by the flame test, and is not free from sulphuric or arsenic acids. Substances consisting chiefly of earths or metallic oxides are treated by triturating forty or fifty milligr. in fine powder with five parts by volume of a previously prepared mixture of four parts by weight of soda with one of silica (as proposed by Berzelius for the quantitative separation of phosphoric acid from alumina), in the agate mortar, transferring it to a soda-paper cylinder, and fusing it in O. F. on coal to a clear bead. The bead is pulverized in the steel mortar, or between paper, and boiled in a small porcelain dish with a sufficient quantity of water. Phosphate of soda and the excess of soda dissolve, while in presence of alumina, silicate of alumina and soda, with other earths or metallic oxides, remain behind. If the substance contained little or no alumina and no sesquixode of iron, a notable amount of silicic acid is dissolved, but this does not affect the determination of the phosphoric acid in the liquid. When the solution is complete, the dish is removed from the flame, and after the undissolved parts have settled, the liquid is either filtered or carefully decanted, with the aid of a glass rod, from the residue into another small dish. If there is reason to suppose that much silicate of soda has been dissolved, it is well to boil the decanted liquid with addition of carbonate of ammonia, when the silica separates in a gelatinous state. After filtering this out, the solution is supersaturated with acetic acid and stirred with some acetate of lead, when, if the phosphoric acid amounts to several per cent., a white precipitate of phosphate of lead is at once formed, which is collected on a filter, dried, and fused in a shallow cavity on coal. If it has been well washed, a white or yellowish globule, with a crystalline surface, is obtained, which behaves like phosphate of lead (p. 244, *et seq.*). It may further be tested with boracic acid and iron.

When the precipitate formed by acetate of lead is so trifling that it cannot be removed without partially destroying the filter, which is to be avoided, as it would then be rendered impure by the silica, of which the filter ash is chiefly composed, a drop of dilute sulphuric acid must be added, so as to produce a mixture of sulphate and phosphate of lead, in such quantity that it may be readily transferred from the filter to the coal. When this is fused B. B., the sulphate is reduced partly to sulphide of lead, which soon volatilizes, and partly to metallic lead, which gradually volatilizes and leaves small globules of phosphate of lead, that can be recognized with the aid of the magnifier, by reason of its characteristic qualities.

When the amount of phosphoric acid is supposed to be very small, a larger quantity of the substance, about 100 milligr., should be fused with five times its volume of the mixture of soda and silica, in two or three portions, and the fused beads then treated as before. This is especially necessary with many iron ores. Any arsenic acid present is reduced and volatilized, while any sulphuric acid forms sulphide of sodium, which goes into solution and yields sulphide of lead, since it is not decomposed by the acetic acid. The sulphide of lead does no harm, however, as it is volatilized on the coal and leaves the phosphate of lead alone.

9. CHLORINE, Cl.

Its occurrence in the mineral kingdom.

Chlorine is always found in nature combined with other elements. The minerals containing it as an essential constituent have been enumerated under potassa, soda, ammonia, lime, iron, lead, copper, mercury, and silver.

Examination for Chlorine,

Including the general blowpipe characteristics of chlorides and chlorates.

Most of the *chlorides* are fusible in the matrass. Those free from water are more or less volatile; the chlorides of the alkalies, alkaline earths, manganese, copper, and some others, are not decomposed; chlorides of gold and platinum are reduced.

On platinum wire and coal the chlorides, even those which withstand the heat in the matrass, are more or less readily decomposed into oxides and hydrochloric acid by the vapour of water in the blowpipe flame, or are reduced, especially on coal. Many are also either entirely or only partially volatilized, and form a coat.

Chlorates. The *chlorates* fuse very easily in the matrass, and when the base is an alkali, alkaline earth, or other strong base, yield oxygen at a red heat, which causes a glowing splinter to burn when introduced into the mouth of the matrass; after strong ignition, continued for a sufficient time, pure chlorides remain. Salts of weaker bases evolve oxygen and chlorine, and leave basic chlorides. On coal the chlorates detonate more violently than the nitrates, and leave neutral chlorides, if the bases are powerful; the feebler bases remain as basic chlorides.

According to Berzelius, chlorine may be detected in its compounds by dissolving oxide of copper in S. Ph. on platinum wire with the O. F., until the glass is opaque, and then causing the substance under examination to adhere to the soft bead, which is then treated with the tip of the blue flame. If chlorine is present, the bead will be surrounded with an intense azure-blue flame of chloride of copper, which volatilizes so long as chlorine remains. A fresh addition of the substance will reproduce this reaction. Bromine is the only other body occurring in minerals which produces a similar flame. By this simple test chlorine can be detected with certainty in earths, oxides, and salts.

When the substances contain little chlorine, or in case of compounds which injure the platinum when heated, a little of the powder is mixed with one-third its volume of oxide of copper, triturated in the agate mortar with a little water, and a few drops of the mixture spread upon coal with the pestle. The mass is dried B. B., without heating it to redness, and then the blue flame is directed immediately upon it, when the azure-blue flame of chloride of copper appears as before. The flame is at first rather greenish-blue, but soon becomes azure-blue. When the compound contains no chlorine, there will be no perceptible coloration. Chlorides, like chloride of silver, which cannot be powdered, must be beaten as thin as possible between paper, cut up with the scissors, and treated on coal with water and oxide of copper as above.

Another method proposed by Berzelius for detecting chlorine in chlorides soluble in water, consists in placing some protosulphate of iron, or sulphate of copper, on a bright sheet of silver, moistening it with a few drops of water, and laying the chloride upon it, when the silver will, after a time, assume a black colour, resembling bronzed ware. According to Merlet, the same test may be applied to insoluble chlorides, after first fusing them on platinum wire with a little soda, so as to form soluble chloride of sodium. The absence of bromine, which produces similar reactions, must previously be ascertained.

10. BROMINE, Br.

Its occurrence in the mineral kingdom.

Bromine has thus far been found in minerals only combined with silver, in *bromyrite* and *embolite* (*vide* silver).

It also occurs in exceedingly minute quantities as *bromide of sodium* or *magnesium*, in certain salt springs.

Examination for Bromine,

Including the general blowpipe characteristics of the bromides and bromates.

Bromides and *bromates* behave like the corresponding chlorides and chlorates in the matrass. On coal the *bromates* detonate with some violence and leave neutral, or, in case of weaker bases, basic bromides. Bromides of potassium and sodium give a white coat on coal (p. 69).

Many bromides are either volatilized or decomposed on platinum wire, or on coal, and diffuse an offensive odour, similar to chlorine. According to Berzelius, they afford with S. Ph. and oxide of copper, or with sulphate of copper on silver, the same reactions as the chlorides; the flame has not a pure azure-blue colour, however, but inclines to green, especially on the edges (p. 78). When all the bromine is gone, the green flame of the oxide of copper alone remains.

To distinguish bromides from chlorides with certainty, Berzelius has proposed to fuse them in the matrass with bisulphate of potassa, when bromine and sulphurous acid are liberated and the matrass is filled with reddish-yellow vapours of bromine, which can be recognized by the similarity of their odour to that of chlorine, notwithstanding the sulphurous acid. *Bromide of silver* forms an exception, as it yields very little bromine, but it may be distinguished from chloride of silver by the asparagus-green colour which it assumes when exposed to the sunlight, after fusion with the bisulphate of potassa (p. 284).

When very little bromine is present, the matrass should be held vertically, after the fusion, so that by looking down through it a thicker stratum of the vapours may be observed than could be seen by looking sideways through the wide part. Chlorine, if present, is also liberated in the gaseous state, but its yellow colour will scarcely be perceptible, if little is evolved. When iodine is also present, the reaction becomes, indeed, uncertain, on account of the violet iodine vapours.

To detect bromine in brine, *e. g.*, the mother-lye from salines, Balard has proposed to pass a current of chlorine through the lye, and then add some ether to it and agitate the whole thoroughly. On allowing it to rest, the ether separates and is coloured hyacinth-red by the dissolved bromine. This ether, on being shaken up with a solution of caustic potassa, loses its colour, and the bromine combines with the potassa, so that after evaporating to dryness the salt can be tested for bromine by means of bisulphate of potassa. Heine has employed chlorine water in place of the gas, to separate bromine from its combinations in saline waters, etc.

11. IODINE, I.

Its occurrence in the mineral kingdom.

Iodine occurs in the rare minerals *iodyrite* (*vide* silver), and *schwartzembergite* (*vide* lead).

It is also occurs with sodium and magnesium in mineral waters, especially such as contain chloride of sodium.

Examination for Iodine,

Including the general blowpipe characteristics of the iodides and iodates.

Most *iodides* can be fused, but not readily volatilized, in the matrass. In presence of water, or an iodate of a weak base, vapours of iodine occasionally result.

The *iodates* are easily decomposed. In the matrass the salts of the alkalies and alkaline earths evolve oxygen and leave slightly alkaline iodides. The other iodates at the same time evolve violet iodine vapours and leave basic iodides, or only oxides. On coal the iodates deflagrate slightly and leave either iodides, or, in case of weaker bases, residues free from iodine. Many iodides behave like the corresponding bromides when treated on platinum wire or coal.

According to Berzelius, iodides tested with a bead of S. Ph. containing oxide of copper, produce an intense green flame.

When iodides are fused in the matrass with bisulphate of potassa, the iodine is partly sublimed and partly fills the matrass with violet vapours, while sulphurous acid is simultaneously evolved. The test is so delicate that small quantities of iodine may be detected in salts, etc., but iodide of silver is only partially decomposed; violet vapours are, indeed, liberated, but most of the iodide of silver collects beneath the fused acid salt to a drop, which retains its yellow colour in the sunlight.

To detect a little iodine in saline waters, which have been nearly freed from chloride of sodium by evaporation, a solution of starch in boiling water is usually employed, with chlorine water, by which an insoluble compound, having a very fine blue colour, is formed. Heine has substituted nitric acid for chlorine water with better success, and proceeds thus: The solution of starch in hot water is stirred into the neutral solution to be tested for iodine, a few drops of nitric acid are added, and the whole again stirred. A very trifling quantity of iodine produces an intense blue colour.

Stein has proposed the following method for detecting very small quantities of iodine in nitric acid and in Chile saltpetre. A suitable quantity of the acid under examination is put into a test tube and a tin rod immersed in it, until red vapours are distinctly visible, after which the tin is withdrawn, a little bisulphide of carbon added, and the whole, after thorough shaking, allowed to stand a few moments. The stratum of bisulphide usually collecting above the acid only has a red colour, unless there is too small an amount of iodine. With traces of iodine the stratum may have only a dark yellow colour, but this turns to a red when the bisulphide is drawn off and a portion of it evaporated by blowing upon it in a porcelain dish.

A method of detecting iodine by the dry way has been proposed by Pasquale la Cava, which is said to be much more certain and sensitive than the wet process with starch. The mass suspected of containing iodine is mixed with a little air-slaked lime and the mixture dried. According to Berzelius, a mixture of carbonate of lime and quicklime, free from water, would be more convenient. The result of the test depends upon the complete absence of water. After very intimately mixing the mass with a little protochloride of mercury, it is transferred to the closed tube, which is then drawn out to a fine tube a little above the mass. Upon heating the mass to redness, iodide of mercury sublimes into the narrow part, where it may easily be recognized by its colour, generally yellow at first, but afterward red. Lime decomposes the protochloride but not the iodide of mercury, which sublimes.

Spring waters containing chlorides of calcium and magnesium afford by evaporation a fluid residue, and some alkaline carbonate must first be added to them, in sufficient quantity to separate the earths, so that a perfectly dry residue may be obtained.

12. FLUORINE, F.

Its occurrence in the mineral kingdom and in metallurgical products.

Fluorine always occurs in combination with other bodies. It forms an essential constituent of the following minerals: Cryolite and chiolite (*vide* soda); amblygonite (*vide* lithia); fluorite and yttrocerite (*vide* lime); wagnerite and chondrodite (*vide* magnesia); topaz (pycnite) (*vide* alumina); fluocerite and parisite (*vide* cerium). It likewise occurs as an unessential ingredient in several other minerals; viz., certain micas (*vide* potassa); certain apatites, pyrochlore and holmite (seybertite) (*vide* lime); hornblende (*vide* magnesia); carpholite (*vide* alumina); zwieselite (triplite) (*vide* manganese). In many smelting processes slags are produced which contain more or less fluoride of calcium, arising from fluorite abundantly disseminated in the ores, or purposely added, and which has not been entirely decomposed by the silica present.

Examination for Fluorine or Hydrofluoric Acid.

When fluorine occurs in trifling quantity with weak bases and a little water in minerals, it is only necessary to heat a small quantity of the substance in the closed glass tube, in which a strip of moistened Brazil-wood paper is inserted. The gaseous fluoride of silicium, expelled by the heat, is decomposed by the vapour of water, and a ring of silica is deposited near the assay, while the escaping hydrofluoric acid colours the paper straw-yellow. This reaction is produced when only three-fourth per cent. of fluorine is present, as in mica.

When no reaction for hydrofluoric acid is obtained, either on the glass or the paper, in the closed tube, Berzelius's test with S. Ph. must be employed. The finely powdered substance is mixed with S. Ph., previously fused on coal and also powdered, and the mixture heated in the open tube, so that the flame may be carried inside of 'the tube by the current of air. Under the solvent action of the S. Ph. upon minerals free from silica, hydrofluoric acid is formed, which passes through the tube and can be recognized both by its peculiar pungent odour and by its effect on the glass, which it attacks and renders dull, especially where any moisture has collected. The escaping air will also turn Brazil-wood paper yellow. In presence of silica, *e. g.* in native silicates and slags, fluoride of silicium is formed, which is decomposed by the water that separates from the products of combustion of the flame. The separate silica dissolves in the water, which condenses on the glass and is gradually evaporated by the hot gaseous products, leaving distinctly perceptible silica behind. When the tube is washed out and dried with blotting-paper, the glass itself occasionally shows dull spots, where it was attacked by hydrofluoric acid. A strip of moistened Brazil-wood paper inserted in the tube before the assay is begun is coloured yellow.

The heat must be sufficient to fuse the mixture, and it may easily happen that a very thin glass tube will soften and contract, so as to interrupt the operation before any result is attained. To remedy this Smithson fastens some platinum foil in the end of the tube with wire so as to form a semicircular tube, Fig. 68; where, however, the platinum is simply inserted into the tube without the wire. The assay is placed on the platinum, and the blast so directed that the products may be driven into the tube. This affords an advantage, inasmuch as the assay is fused without coming into contact with the glass.

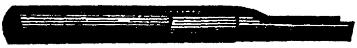
Fig. 68.

According to Merlet, silicates containing not too little fluorine may be tested by fusing the fine powder with equal parts of melted bisulphate of potassa (according to Berzelius, with four times its weight), in the closed tube, either with the spirit-lamp or the blowpipe, until sulphuric acid begins to escape. The substance should be heated from above downward, otherwise the whole mass may be driven out. The empty portion of the tube is thus more or less thickly coated with silicic acid, which separates from the gaseous fluoride of silicium. The tube is then cut off just above the fused mass, washed out with water and dried with blotting-paper. In presence of much fluorine the glass is quite dull throughout, but when there was a trifling amount it only shows dull spots here and there. This test is, however, inferior to that in the open tube with S. Ph., for detecting very trifling quantities of fluorine.

13. CYANOGEN, Cy. COMPOSITION=C^2N.

Its occurrence in metallurgical products.

When iron ores are smelted in blast-furnaces with charcoal, the carbonate of potassa in the coal is liable to form *cyanide of potassium;* while the carbonate is reduced to potassium, with formation of carbonic oxide, cyanogen is apparently produced from the coal in contact with the potassium and from the nitrogen of the blast, and this combining with the potassium either escapes in a gaseous state from the throat, or, when the furnace is worked with a closed breast, through the openings, above and below which it condenses as a white or gray salt. When the gases from the throat are collected for any purpose, the same salt occasionally collects in the conduits, but is more or less impure from coal and ore dust. In contact with moist air this salt is liable to partial decomposition, and generally consists of mixed cyanide of potassium, K Cy, cyanate of potassa, $K\ \dot{C}y$, and carbonate of potassa, $\dot{K}\ \ddot{C}$; in presence of carbonate of potassa it also usually contains a corresponding ammonia salt, as well as more or less disseminated carbide of iron and coal.

Cyanogen also occurs combined with titanium and nitride of titanium in small crystals and amorphous masses, in the cadmia of certain iron blast-furnaces (*vide* titanium).

Examination for Cyanogen,

Including the general blowpipe characteristics of cyanides.

Cyanides, excepting those of the alkalies and alkaline earths, heated to low redness in the closed tube or matrass, are decomposed, becoming charred, and evolving cyanogen, ammonia, water, and nitrogen. Ag Cy is decomposed into metallic silver, or carbide of silver and cyanogen; Hg Cy separates into cyanogen and mercury, which sublimes, and a black powder is left behind (paracyanogen—Cy N). The anhydrous cyanides of the alkalies and alkaline earths suffer no change at a red heat, and cyanide of potassium may even be brought to a moderate white heat.

On coal and in the platinum spoon all the cyanides are decomposed, but those of the alkalies slowly, and the liberated cyanogen is consumed.

The wet way is best suited for detecting cyanogen in the salt above referred to, as being formed in smelting iron or with charcoal. A little of it is dissolved in water and decanted after the residue of coaly matters and particles of iron has settled. The solution is then acidified with hydrochloric acid, when a little prussic acid is evolved, and effervescence ensues if carbonate of potassa was present. To the acid solution a few drops of a solution of proto-sesquioxide of iron (magnetite, or siderite ignited in the matrass) in hydrochloric acid are added and afterward solution of potassa in drops, when the presence of cyanogen will immediately afford Prussian blue. The test may also be made by testing a part of the dissolved salt for carbonic acid with hydrochloric acid alone, and adding to the other portion a few drops of the solution of proto-sesquioxide of iron, when a grayish-green precipitate is produced. By then adding a slight excess of potassa solution, shaking it up and adding hydrochloric acid to strongly acid reaction, the Prussian blue is obtained on agitating it anew. The latter method is to be preferred when little cyanogen is present, as none of it is then lost by the formation of prussic acid.

SECTION III.

QUANTITATIVE BLOWPIPE ASSAYS.

I. Preparation of the substances to be quantitatively examined for certain constituents.

In quantitative assays with the blowpipe, just as by the wet process, the substance to be examined must undergo certain preliminary operations, which are limited chiefly to drying, and, in case of mixed substances, selecting the best possible average sample. Friable substances containing mechanically combined water should be dried at a temperature of 100° C., and then pulverized in an agate mortar. If the substance is brittle, but not friable, it is broken as fine as possible between paper on an anvil; if malleable, it is beaten between paper into a thin sheet and then cut up with the scissors.

Ores dressed on a large scale, even when they have not been especially dried, usually appear dry while still containing several per cent. of mechanically combined water; the same ores also absorb moisture again when kept in damp places in unclosed vessels after being dried. A quantity of these, more than sufficient for two assays, must be dried in a porcelain dish over a lamp, at about 100° C., and the dry ore pulverized in an agate mortar. Care must be taken to avoid too high a temperature when drying ores containing compounds of sulphur or arsenic, since otherwise, roasting, with partial decomposition of the ore and alteration of weight, may result.

Minerals and metallurgical products, which are almost always received for analysis in a dry state, are broken up between paper on an anvil, or in a steel mortar, and when friable, pulverized in an agate mortar. It is safest to prepare from the substance to be assayed eight or ten times the amount of powder needed for one assay, except in cases of pure crystals, or fragments of minerals or metallurgical products, since, when too small a quantity is used, there is no certainty that a proper average has been attained, as regards the proportion of metal present.

For example, when too small a quantity of a rich dry-stamped silver ore, which may be a mixture of real silver ore and substances containing no silver, is taken, one may have too many rich portions, or too many poor portions, which will give a very different proportion of metal from that contained in the whole. From an ore prepared on the large scale, therefore, a quantity of at least thirty grammes should always be taken from various parts of the heap, mixed if possible in an iron mortar and made somewhat finer, and from this the portion required for the blowpipe assays, from eight to ten blowpipe centners, taken, dried according to the preceding directions, and rubbed quite fine in a mortar.

II. Detailed description of the quantitative assays with the blowpipe.

1. THE SILVER ASSAY.

THE blowpipe assay for silver, first proposed by Harkort, and described by him in a work that appeared in Freiberg, in the year 1827, is one of the most important assays that can be made with this instrument. It is possible not only to detect in a short time the silver in any ore, mineral, or product, but also to determine its amount quantitatively with all needful accuracy. Regard must, however, be paid to the other substances besides silver, with which we have to do, and we must classify the mineral and metallic bodies, together with the artificial products, in order to determine the silver in them, and each of these classes must be assayed by a suitable method.

They are classified into :—

A. Ores, minerals, and products in which the silver is especially combined with non-metallic bodies; and these further into :

a. Those containing volatile constituents, viz., sulphur and arsenic, as well as chlorine, bromine, and iodine, in greater or less proportion, or such as are wholly free from them and can be decomposed by fusion on coal with borax and test lead.

b. Those containing compounds which cannot be decomposed by fusion with borax and test lead alone.

c. Those consisting of metallic oxide that are readily reduced on coal.

B. Metallic compounds (*alloys*); these are:

a. Those in which silver is the chief constituent, or in which gold occurs with the silver.

b. In which copper or nickel forms the prevailing constituent, while silver is only a minor one.

c. In which lead or bismuth is the chief constituent.

d. In which tellurium, antimony, or zinc is the chief constituent.

e. In which tin is the chief, or else only an accessory constituent.

f. In which mercury is the prevailing constituent

g. In which iron or steel is the chief constituent.

A. Assay for Silver in Ores, Minerals, and Metallurgical products in which the silver is especially combined with non-metallic bodies.

a. Substances which contain volatile constituents, viz., sulphur and arsenic, as well as chlorine, bromine, and iodine, in greater or less proportion, or are entirely free from them, and can be reduced by fusion on coal with borax and test lead.

Here belong, among ores dressed on a large scale, such as contain more or less *pyrites, chalcopyrite, mispickel, stibnite,* and *blende,* as well as the minerals above mentioned; further, all the so-called *Dürrerze,* consisting chiefly of earthy ingredients, and containing only a small portion of actual silver ores; all ores in which the silver occurs in combination with selenium or sulphur and other selenides or sulphides, as well as with chlorine, bromine, and iodine; further, all copper ores in which the copper is combined with selenium or sulphur; lead ores in which the lead is present as selenide or sulphide of lead; further, roasted argentiferous lead and copper ores; all silver ores and metallurgical products roasted with salt, for the purpose of amalgamation or extraction, and the residues from amalgamation or extraction; finally, among metallurgical products, *Rohstein*, lead and copper matts, cadmia, flue-dust, lead and cobalt speisses, hearths from cupelling and refining silver, all sorts of argentiferous slags, and also the silver scraps of the gold and silver smiths.

1. WEIGHING AND CHARGING THE ASSAY.

Ores consisting of a mixture of rich silver ores and earthy parts, which usually yield in analysis varying amounts of silver, are best weighed out in two or three portions of 1 centner =100 milligr. each; on the contrary, poor silver ores and crystallized minerals, as well as products which differ very little or not at all in richness, are generally weighed out only once.

The weighed portion is transferred to a mixing capsule, into which the scale-pan is cleaned with a brush, and borax-glass and test lead are then added. The quantity of borax-glass is regulated by the fusibility and amount of the substances to be converted into slag. The small spoon (Fig. 50) heaped full and containing about one decigr. or one ctr. of borax-glass, is sufficient for an assay of difficult fusion, but if during the fusion the assay seems too refractory, a small portion of this flux can be added. Less is needed with very fusible ores, or in general, such as have no earthy admixture, but consist only of metallic sulphides which combine readily with the lead, and are less oxidizable than it. For these a spoon but slightly heaped, containing from one-half to three-quarters ctr., is quite sufficient. With assays containing a considerable proportion of earthy ingredients, or much iron, cobalt, or tin, the spoon must always be heaped full.

The quantity of test lead is regulated by the presence of other metals in the assay substance.

If it is a substance containing not more than seven per cent. of copper or ten per cent. of nickel, five ctrs. of lead are used for one ctr. of assay powder, being measured in the test lead measure; if the substance contains more than seven per cent. of copper or ten per cent. of nickel, the amount of lead must be proportionally increased. The presence of cobalt is less to be regarded, as this metal is easily slagged off with borax. As we cannot always know beforehand how large an amount of these metals is present, it is better to use too much lead than too little, since with too little lead the separation of the copper from the silver is not thorough, while the cupellation of lead rich in nickel is almost impossible.

The following minerals and products, containing partly copper and partly nickel, must therefore be charged with the indicated amounts of test lead :—

1	ctr.	Chalcocite,	containing about	80 °/o	Copper,	with	15	ctr.	test	lead.
1	"	Covellite	"	65—66	"	"	12	"	"	"
1	"	Bornite	"	55—60	"	"	12	"	"	"
1	"	Tennantite	"	48—50	"	"	10	"	"	"
1	"	Zincfahlerz	"	40—41	"	"				
1	"	Tetrahedrite	"	30—40	"	"				
1	"	Chalcopyrite	"	30—34	"	"				
1	"	Stromeyerite	"	30—31	"	"				
1	"	Stannite	"	29—30	"	"	7	"	"	"
1	"	Eucairite	"	23—25	"	"				
1	"	Bournonite	"	12—13	"	"				
1	"	Copper Matt	"	30—50	"	"	10	"	"	"
1	"	" "	"	50—70	"	"	12	"	"	"
1	"	Lead speiss	"	10—40	" Nickel, Cobalt, and Copper	"	10	"	"	"
1	"	Cobalt speiss with		40—50	" Nickel and Cobalt	"	10	"	"	"

When the substances have been most thoroughly mingled in the mixing spoon, with the aid of the ivory spoon handle, the charge is poured into a soda-paper cylinder, made as directed. For this purpose the cylinder is held between the thumb and index finger of the left hand and the mixing spoon with the same fingers of the right hand, while the closed end of the cylinder rests on the middle finger of the left hand. The lip of the capsule is then inserted into the paper cylinder, which is slightly inclined to one side, as far as seems necessary for safely pouring in the charge, the paper being pressed against the lip with the finger and thumb, so that the capsule cannot fall on removing the right hand. The charge is then caused to slide gradually into the cylinder by gently tapping on the outside of the capsule with the brass forceps, and any adhering dust is brushed into the paper. While the lower end of the cylinder still remains resting upon the middle finger of the left hand, the upper empty end is pressed out flat and then rolled up from the top downward; the cylinder thus far closed is placed with the lower end on the extremity of the left thumb, and the ends of the part that was rolled together are bent upward and toward each other, making the whole perfectly tight. While wrapping up the charge, care must be taken that the lower end of the cylinder does not open or the paper tear in any part, thus occasioning mechanical loss.

2. THE FUSION OF THE ASSAY.

The fusion of a silver assay is performed on coal with the blowpipe flame. In the cross section of a good piece of charcoal, near one corner, a deep cylindrical hole is bored with the square borer, the diameter of which is about one-third more than that of the paper cylinder; or a coal crucible is used, as shown in Fig. 69, A. The latter must also be bored out as deep as necessary, and the hole widened from above with the knife, so that the flame can be directed between the assay and the inside of the crucible down to the bottom, and the assay thus readily melted. The cylinder is set in the cavity with the end last closed above, and is pressed down firmly. The assay is now inclined toward the flame, and a pure, but at first not too violent, reducing flame directed upon it, so that it almost covers the upper part of the paper cylinder.

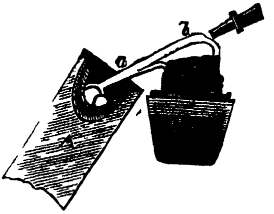

Fig. 69.

The soda-paper is indeed charred in a few moments, but the ashes are not disturbed until the borax-glass has already fused together the separate particles of ore, so that they cannot be blown away. When the ashes of the paper burn away, exposing a part of the charge as a fluid slag, mixed with melted globules of lead, the whole assay is covered with a strong, but pure, reducing flame, which has an inclination of 30° to 35°, as shown in Fig. 69.

During the time that this flame is used, portions of the sulphur, arsenic, antimony, zinc, etc., volatilize, but the greater part of them, as well as several of the metals still combined with sulphur and arsenic, join with the lead and melt with it to a button. The earthy ingredients, on the other hand, and the metallic oxides of difficult reduction, with a small part of the non-volatile and easily oxidizable metals, which become in part oxidized by the first action of the heat, fuse with the borax to a slag. With compounds of silver and chlorine, bromine, or iodine, which are decomposed by the lead, vapours of chloride, bromide, and iodide of lead are seen to pass off.

Although it generally seems, after a short time, as if the slag was quite free from lead globules, this must not be regarded as satisfactory, since beneath the well-fused slag there are often unmelted portions of the charge, which can only be affected by the blowpipe flame when this is directed between the slag and coal against the bottom of the crucible, while the charcoal, or the clay cylinder with the crucible, is turned during the blast and held inclined toward another side, until the assay has changed its position and *turned over*. In this turning over, which should take place even with the most fusible assays, the bottom of the paper cylinder, being all that remains of it, rises from below, and comes in a charred state to the top, or to one side. To destroy the ashes of the paper the assay must then be so held toward the flame that only that part of the slag where there is no paper is covered by it, when the air instantly rushes in and the ashes are consumed. When the position of the slag *c*, which has been covered with the reducing flame *ab*, Fig. 69, has been altered several times with regard to the lead button *d*, and the slag finally appears quite fluid and free from lead globules, it is also certainly free from silver. The R. F. is changed to a moderate O. F., which is directed against the lead only, and at a somewhat greater distance. The above-named volatile metals, with the sulphur, are now driven from the lead, while some of the easily oxidizable metals, as iron, tin, and cobalt, with a small part of the nickel and copper, are oxidized and combine in this state with the slag; only the silver, with the greater part of the copper and nickel, remains with the lead. In substances containing much arsenide of nickel it is difficult to destroy this combination, which does not combine readily with the lead, but remains on top of it, and must be treated a long time with the O. F. to oxidize and slag-off all the nickel and arsenic. Since, however, this compound readily yields its silver to the lead, no loss of silver need be feared, even if the arsenide of nickel is by no means entirely decomposed, and in many cases it may even be mechanically separated from the lead with advantage after the assay has cooled.

When the volatile parts of the assay are nearly gone, a part of the lead is oxidized, and with it a trace of the silver, which is, however, very trifling. Both oxides are taken up by the slag, but as this is always in contact with the coal, a portion of the dissolved oxide of lead, exceedingly poor in silver, is reduced at the points of contact, occasioning a bubbling in the slag. The slag has now lost its spherical shape and has spread out, and the reduced globules of lead, first appearing on its borders, are gradually carried toward the argentiferous lead button by the motion of the slag, and unite with it. When the volatile matters are quite gone, the lead button begins to oxidize more rapidly and assumes a rotary motion, while the bubbling in the slag is livelier. On observing these signs, the coal is inclined a little, so that the lead button may go to one side in case it is quite surrounded by slag, the blast is stopped, and the assay allowed to cool upon the inclined coal. When there are few or no constituents that require to be volatilized by an oxidizing blast, a short treatment only with the O. F. is needed, after the fusion of the silver particles with the lead and the conversion into slag of the earthy portions and the metallic oxides of difficult reduction.

The fusion may be regarded as completed if, after the assay is cold, the argentiferous lead or *silver-lead*, has a white colour; if, however, it appears black, this depends, in case of a substance free from copper, upon some sulphur or antimony remaining in it; with a substance containing copper, on the other hand, either upon the copper alone, or upon this and both of the others at the same time. Sulphur and antimony can be removed in both cases by treating the assay again with the O. F., but the copper can only be separated together with the lead during the cupellation. In assaying any substance for silver, therefore, a white lead cannot be expected if more than a trifling amount of copper is present, and we can only assume that the sulphur is all removed when the lead has been in rather strong rotary motion for at least one minute. The complete removal of volatile bodies from the lead by an oxidizing fusion is necessary for two reasons; first, because the impure lead is generally brittle, and some of it may easily be lost in breaking off the slag; and, secondly, because it is liable to sputter on the cupel, especially when containing sulphur.

When the assay is cold, the slag and lead are lifted out of the crucible with a knife, laid on an anvil, and the slag separated as much as possible with a few strokes of the hammer, after which the lead is held with the forceps and beaten into a cube. Should a small button of arsenide of nickel adhere to the lead from an assay rich in nickel, as mentioned above, an attempt must be made to separate it here, that it may not impede the cupellation.

Among the ores, minerals, and products to be fused according to the preceding method, the most infusible are pyrites, mispickel, certain nickel and cobalt ores, and such *Rohsteins* as consist chiefly of sulphide of iron; the other substances in this class fuse, for the most part, very easily, even when mixed with refractory earthy matters.

No right result would be obtained if, as beginners frequently do, the attempt were made to perform the fusion of a silver assay with the O. F., since a considerable amount of lead would be oxidized at the very beginning, the oxide would dissolve in the borax, and being reduced by the coal, would form fresh globules of lead, which would abstract a portion of the silver still remaining in the slag. On attempting, after a few moments, to collect these separated globules by varying the position of the chief button in the slag, which would have spread out to a great extent over the coal, new ones would constantly form in their place, which could not be distinguished from those containing silver. The spreading of the slag also renders the complete fusion of the charge at the bottom of the crucible difficult or quite impossible, and an assay in which this mistake has been committed should be regarded as useless. The time required for the above fusion depends upon the assay to be treated. If it contains a large amount of volatile substances, or such as must be slagged off, eight to ten minutes are necessary; if but little, about five minutes. When several assays are to be made, the second can be fused while the first is cooling; then the third is taken up, and so on, until all the assays weighed out have been fused. The assays must be arranged according to their number, to avoid confusion. When the several assays have been fused, the first ones will be cold, and can then be freed from slag in their proper order. When but one assay is to be made, the cupel for it can be formed, as will be described under cupellation, while the silver-lead is cooling.

3. CUPELLATION OF THE SILVER-LEAD OBTAINED BY THE FUSION.

It is well known that the cupellation of the silver-lead is an oxidation occurring at a red heat with access of air, by which the lead with other oxidizable metals is separated from the silver, which oxidizes with difficulty. In the blowpipe assay this oxidizing or cupelling process is divided into two periods, viz., the *scorification* and the *fine-cupellation*. This division is necessary because it is not possible to separate a large quantity of lead from the silver in one period, so that the latter may remain in the form of a pure round button, as in the muffle assay.

We will, therefore, now describe the first period, or

THE SCORIFICATION.

This is the easiest operation in the whole silver assay. A cupel of sifted bone-ash is struck in the cupel mould placed on the stand, and the bone-ash heated with the O. F. in all parts as strongly as possible, so as to remove any remaining hygroscopic moisture. If this heating is omitted, the steam escaping during the fusion is liable to cause the lead to sputter and be lost. After heating the cupel, the lead is placed in the middle of it with the forceps, and brought into fusion with a rather strong O. F., so that the oxidation of the rotating lead begins. During this operation the cupel is inclined slightly backward from the lamp, and the result is effected most rapidly by allowing the point of the blue flame to act directly on the lead. When the lead contains much copper or nickel, the period of fusion, before the oxidation begins, is somewhat prolonged, since the copper makes the lead less fusible, while the nickel separates as the lead begins to oxidize, covering the whole surface with an infusible coat, and causing a difficult cupellation, or, with too little lead, entirely preventing the operation. In the latter case, a piece of pure lead of from two to four ctrs., according to the thickness of the crust, must be added to the silver-lead in the cupel, and then only is the cupellation possible.

Persons not accustomed to using the blowpipe sometimes experience inconvenience in not immediately causing a large button of silver-lead to oxidize, or else it *freezes* during the process, becoming covered with a coat of oxidized lead which they cannot easily drive away. In this case a somewhat stronger blast must be used, and the lead touched directly with the point of the blue flame, and after this has acted uninterruptedly on one point of the lead button, the lead will soon begin to oxidize again.

When the lead has been brought to the proper temperature for cupellation, the tip of the blowpipe is thrust farther into the flame, so as to produce a fine blue point, *a*, Fig. 70, which is directed at an angle of about 30° upon the oxidizing lead, so that this is kept at a moderate red heat, but is only touched by the outer flame, and on no account by the blue flame. The surrounding air thus has free access, while the lead (and copper) absorb a portion of the oxygen from it and become oxidized. The *oxide formed* flows from the upper surface of the lead to the border, exhibiting prismatic colours caused by the interference of light, and solidifies on the cupel behind the button to a firm mass called *litharge* (*Fig.* 70, *c*), which has a reddish-yellow fracture after cooling, if free from other oxides. When the lead contains very much silver, the prismatic colours are less distinct, and thus a large proportion of silver is already indicated; if it contains copper, the colour of the solid litharge is nearly black.

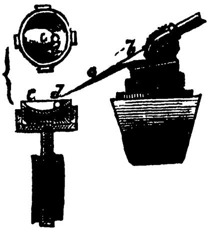

Fig. 70.

The cupellation must proceed at neither too high nor too low a temperature. If it is too hot, the lead begins to vaporize, and some silver may easily be mechanically carried off, especially if the lead is rich in silver; moreover, the litharge does not cool upon the cupel, but sinks into it, by which again a part of the silver is lost, since the surface of the lead is covered with too little fused oxide of lead, and the silver has a chance to become oxidized. If the cupellation is carried on too "cold," and the temperature is not high enough to continue the oxidation of the lead, the latter becomes covered with too much litharge, the motion of its surface ceases, and it *freezes*.

This mistake is less injurious than having too high a temperature, since the frozen assay can be immediately made to oxidize again by a somewhat stronger flame, without loss of silver; but this must not occur often in the same assay.

When the scorification proceeds at the proper heat, which cannot be so clearly described as perceived by practice, the litharge collects around and chiefly behind the lead, and solidifies. After a quantity of it has collected, and the lead in its midst has too little surface exposed, the cupel is gradually brought into another position without interrupting the operation, so that the lead, by reason of its weight, may move to the side of the litharge and expose a greater surface for oxidation. The lead having finally decreased so much that, in an assay not very rich in silver, it is only the size of a mustard-seed (Fig. 70, *d*), and in case of a rich assay, about two or three times as large, the cupel is removed from the flame by degrees, so that the lead button may very gradually solidify in the litharge. The button will indeed always be somewhat raised by the contraction of the cooling litharge, but if too hastily drawn away from the flame, the still soft button will be too violently driven out by the solidification of the litharge, and a spattering of the lead, with loss of silver, may easily occur.

Notice must here be taken of a phenomenon which sometimes appears at the close of the scorification of a lead button very rich in silver. When such lead has been so far oxidized that it consists of about six or seven parts of silver to one part of lead, and allowed to cool slowly in the litharge before a gradually diminishing blowpipe flame, a grayish-white, easily friable mass is forced out from the solidifying lead, which is always very rich in silver. It appears to be a sub-oxide of lead mixed with metallic silver, and is probably to be regarded as a phenomenon allied to the sprouting of silver, which will be mentioned under the fine-cupellation. If unnoticed, the greater part of this mass falls off in separating the lead from the litharge, entailing a not altogether unimportant loss of silver. This evil can be remedied by treating the silver button containing lead immediately on the cupel with the R. F., or fusing with it a small piece of test lead, when the whole unites into a button, which cools with a clean surface. If, therefore, rich ores or products are to be assayed, it is always well either to continue the scorification only so far that the lead still forms more than the sixth part of the silver, or, as this cannot always be known beforehand, not to stop the process until the silver is nearly free from lead, in either of which cases the phenomenon will not appear.

After the scorification, the lead button, which is in, or surrounded by, the litharge, is taken out together with it, and when cool is freed from all adhering litharge, which is very easily done by laying the whole on an anvil and pressing off the fragile litharge from around the button with the broad face of the hammer, without touching the button; any remaining litharge may then be removed by a few strokes of the hammer.

THE FINE-CUPELLATION.

This requires more care and practice than the preceding operation. The bone-ash remaining from the scorification which is not permeated by oxide of lead is broken up with the small iron spatula, covered over with enough elutriated bone-ash to fill the cupel mould, and after placing on it the proper stamp, it is struck with a few blows of the hammer into a cupel for the fine-cupellation. This is then thoroughly heated as before, and if any cracks form, or portions of the bone-ash become loosened during the heating, from moisture remaining in the bone-ash, the fault can be at once remedied by placing the clean stamp upon it and striking it once or twice gently. For this purpose the mould must of course be removed from the stand to the anvil.

The lead button, *d*, Fig. 70, is then placed with the forceps upon the cupel, so that it may lie nearer the left-hand edge than the middle (Fig. 71), that any adhering substance may remain at the edge when the button moves toward the middle, and may not prevent the formation of a spherical silver button during the fine-cupellation. The cupellation is then carried on, observing the following directions:—First, the cupel is brought near the lamp flame, with the stand so much inclined in an opposite direction, that the lead button, which is on the further side, cannot roll down before it has become fused. The button is then heated with the O. F., directed downward as much as possible, until it fuses and begins to oxidize. As soon as this takes place, the stand is gradually brought into a vertical position and the flame *ab* directed at an angle of 40° to 45° upon the cupel, toward the middle of which the button is moving, so as to heat the bone-ash directly about the button and keep it as much as possible in a constant glow around it. This is best effected by moving the cupel slowly around in a small circle before the blowpipe

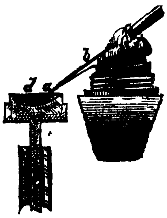

Fig 71.

flame, the direction of which remains unaltered, at the same time inclining the stand as required toward the flame, and giving it, if necessary, a slight rotary motion. Without touching the assay with the flame, a strong enough heat must be imparted to the bone-ash to keep the assay in oxidation without allowing it to become quiet, or to freeze. Should this happen, the solidified lead must be brought for a moment nearer the flame, so as to cause it to oxidize, and the cupel then immediately moved slowly about in a circle before the flame again. The cupellation proceeds better, the drier the surface of the bone-ash; that is, the more perfectly the litharge sinks into it. If the bone-ash is not strongly enough heated, it becomes covered with a thin coat of litharge, in which the button begins to swim about rapidly; and even if this does not ruin the assay, it is still very difficult to detach the silver button from the cupel, and thus an uncertain result is caused.

It is not indispensable, and in case a large amount of lead remains with the silver not possible, to complete the fine-cupellation on one spot of the cupel, but the button may just as well be allowed to roll from one place to another, only keeping the bone-ash around the button at a red heat without touching the latter with the flame. When the last portions of lead have been oxidized from a lead button poor in silver, the rotary motion of the remaining silver button may cease without a change of colour; the heat is then raised to remove entirely the last thin coating of litharge, which separates with the most difficulty, and the silver button is allowed to cool slowly by gradually removing it from the flame. It can be examined with the glass, to see whether it has the pure silver colour, with a bright surface, or whether it requires further heating. If the lead is rich in silver, a play of colour is seen about five or ten seconds before the "brightening" of the pure silver, while the last portion of lead is separating as litharge. Colours appear, similar to those in the scorification, but they are much finer, on account of the thinner coating of litharge which reflects better and causes more perfect interference of the light. They also vanish entirely as soon as the silver is pure.

So long as the fine prismatic colours appear, the cupel should be moved about in a circle before the oxidizing flame, so that the metallic button is nearly touched by the tip of the blue flame and is driven from one point to another, while the blast must not cease until the surface of the silver is quite free from litharge, which can be very well seen with a rich assay. As soon, however, as it shows a clean surface, the assay must be very gradually removed from the flame and the silver allowed to cool slowly.

When a large silver button is heated for some time after it has brightened, some silver may easily volatilize, as can be seen from the rose-coloured coat that forms on the cupel; moreover, here and there on the bright surface of the fluid silver button dull spots appear near each other, having the appearance of foreign substances, which finally form a crust, and have a dull silver-white colour when the button is cold.*

The silver button must be slowly cooled to prevent the so-called *sprouting*, a phenomenon due to the absorption by the silver of a small amount of oxygen during the cupellation, and this escaping again at the moment of solidification may easily cause some loss.

In cupelling silver-lead containing such a quantity of copper that it cannot be completely oxidized at the same time with the lead, the silver button generally spreads out during the brightening, and although it appears white after cooling, is often anything but free from copper. Such a button must be melted immediately, while still on the cupel, with one centner of fused test lead, or if so small as to require measuring on the scale, with one-fourth to one-half ctr., and cupelled fine on another part of the cupel, so that it may become round and quite pure. It is better to perform the final cupellation of a very cupriferous lead in this way than to add at the start as much lead as is necessary for the perfect separation of the copper, since in many cases almost twice as much test lead would then be required and the fusion and cupellation would be rendered more difficult. If in the scorification the proportion of copper oxidized with the lead was as great as in the succeeding fine-cupellation, the copper could be separated by the first fine-cupellation; but as this is not the case, the copper can only be removed by a second refining cupellation with a small quantity of test lead.

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* These appear to be a combination of silver and oxide of silver, and may probably be regarded as over-refined silver, analogous to over-refined copper.

Slight obstacles sometimes arise in the fine-cupellation of the silver-lead, which if disregarded may exercise a very bad influence in determining the weight of the silver button on the measuring scale.

1. In spite of all care a bit of bone-ash or something else may adhere to the lead, and if the operation were continued without regard to this there would be danger of the silver button adhering to it, or, if very small, getting under it, and it would in any case receive a very irregular shape. It is then better to interrupt the cupellation. Lay a little piece of fused test lead near the button and melt the two together, after which they are again cupelled. By this means the mass of lead is increased, and when the cupel is inclined to one side has weight enough to break away from the adhering object and move to another part of the cupel, on which it can be cupelled perfectly fine.

2. When the necessary practice in cupellation is wanting, too low a heat is sometimes used and the litharge surrounds the button as in the scorification, instead of sinking into the cupel. The cupellation must then be stopped, and the button, if large enough, separated from the litharge with the forceps, or if already too small, it must be melted with a small piece of test lead (50–60 milligr.), and on cooling detached from the litharge; in both cases the fine-cupellation is completed on a newly-made and heated cupel.

3. Sometimes, on account of too low a temperature of the cupel, the silver button remains surrounded during the brightening by a little litharge, which has not sunk into the bone-ash, and the button, although apparently pure, cannot be easily separated from the adhering litharge in a clean state. The button with the litharge must then be strongly heated with the O. F., at some distance, until all the litharge has sunk in, leaving behind the pure button, which is allowed to cool slowly.

TA

SHOWING THE LOSS WHICH OCCURS IN CUPELLING

Weight of THE SILVER BUTTON OBTAINED BY CUPELLATION.	The assay contains the following amounts of			
	80 to 99 °/o.	60 to 79 °/o.	30 to 59 °/o.	10 to 29 °/o.
	And has been charged and cupelled			
	16 ctrs. lead.	13 ctrs. lead.	11 ctrs. lead,	8 ctrs. lead.
	The cupellation loss suffered by the			
Milligrammes.	Milligr.	Milligr.	Milligr.	Milligr.
99·5 to 99·75	—	—	—	—
90·	—	—	—	0·83
80·	—	—	—	0·75
70·	—	—	0·82	0·68
60·	—	—	0·74	0·61
50·	—	—	0·65	0·54
40·	—	0·62	0·55	0·46
35·	—	0·57	0·50	0·42
30·	—	0·51	0·45	0·38
25·	—	0·45	0·40	0·34
20·	0·45	0·39	0·35	0·29
15·	0·37	0·32	0·28	0·23
12·	0·32	0·26	0·23	0·19
10·	0·27	0·23	0·20	0·17
9·	0·25	0·21	0·18	0·16
8·	0·22	0·18	0·16	0·15
7·	0·20	0·16	0·14	0·13
6·	0·17	0·14	0·12	0·11
5·	0·14	0·12	0·11	0·10
4·	0·11	0·10	0·09	0·08
3·	0·09	0·08	0·07	0·06
2·	0·07	0·06	0·05	0·04
1·	0·05	0·04	0·04	0·03

DETERMINATION OF THE WEIGHT OF SILVER BUTTONS OBTAINED BY THE ASSAY.

If too large for the ivory scale, the silver button is raised from the cupel with the steel forceps, held firmly and all adhering bone-ash removed with a stiff brush, after which it is weighed with the assay balance. If the button is so small

BLE

SILVER WITH VARIOUS AMOUNTS OF TEST LEAD.

copper 7 to 9 °/o with :	THE ASSAY CONTAINING NO COPPER, OR LESS THAN 7 °/o, HAS BEEN CHARGED AND CUPELLED WITH THE FOLLOWING AMOUNTS OF LEAD.				
6 ctrs. lead.	5 ctrs.	4 ctrs.	3 ctrs.	2 ctrs.	1 ctr.
silver present in the assay is:					
Milligr.	Milligr.	Milligr.	Milligr.	Milligr.	Milligr.
——	0·50	0·45	0·39	0·32	0·25
0·69	0·47	0·42	0·36	0·29	0·22
0·64	0·44	0·39	0·33	0·26	0·20
0·58	0·40	0·35	0·29	0·23	0·18
0·52	0·36	0·30	0·26	0·20	0·16
0·46	0·32	0·26	0·23	0·17	0·14
0·39	0·27	0·22	0·20	0·15	0·12
0·36	0·25	0·20	0·18	0·13	0·11
0·32	0·22	0·18	0·16	0·12	0·10
0·29	0·20	0·16	0·14	0·10	
0·25	0·17	0·14	0·12		ETC.
0·20	0·15	0·12	0·10	ETC.	
0·17	0·13	0·11			
0·15	0·11	0·10	ETC.		
0·14	0·10				
0·13	0·09	ETC.			
0·12	0·08				
0·10	0·07				
0·09	0·06				
0·07	0·05				
0·05	0·04				
0·04	0·03				
0·03	0·02				

that its weight can be better determined on the scale than the balance, it must be carefully detached from the cupel with the small brass forceps, so that it may retain its shape, while as little bone-ash as possible adheres to it. If too strongly pressed, its diameter will be altered, and if any bone-ash adheres to it, the button cannot be accurately measured when placed on its flat side. The best method of proceeding is as follows:

First place the cupel mould upon the anvil, then carefully insert the sharp end of the small iron spatula, or the point of a small knife, between the button and the bone-ash and detach it from the cupel, holding it meanwhile with the small forceps, after which it is cleaned by rolling it between paper underneath the finger on the table, and placed upon the scale. The liability of silver to oxidize causes a loss, called the *cupellation loss*, in cupelling argentiferous lead in the muffle. The same loss occurs in the blowpipe assay, not only in the fine-cupellation, where the litharge sinks into the bone-ash, but also in a less degree during the scorification and during the fusion of the ore, while the assay is subjected to the O. F. It is, however, less than that which occurs in the muffle assay, where all the litharge formed in cupelling the lead must sink into the mass of the cupel.

With only one per cent. of silver this loss is scarcely perceptible on the balance, but it becomes appreciable as the button to be weighed grows larger, and if reckoned by percentage it increases again as the button becomes smaller; it also alters as the quantity of lead to be cupelled increases or diminishes, but remains constant in other respects, for each separate proportion of silver, if the same quantity of lead is always used and the proper temperature employed in the cupellation. Plattner has sought by careful experiments to ascertain the loss that occurs, with a proper temperature for the cupellation, for every weighable amount of silver down to one per cent., with varying quantities of lead. The tabular arrangement of the values found is given on pp. 376, 377. Since, according to p. 363, cupriferous silver ores and minerals are charged with 5, 7, 10, 12, and 15 ctrs. test lead, according to the amount of copper they contain, while the silver button obtained by their cupellation can only be completely refined by adding one more centner of test lead, their cupellation loss is given at once for the whole amount of lead, viz., 6, 8, 11, 13, and 16 ctrs.

If the blowpipe balance is so delicate as to indicate 0·05 milligr., the cupellation loss can be reckoned with both decimal places, and for silver buttons weighing between 70 and 60, or 50 and 60, etc., milligr., it can be reckoned from the difference. For instance, from one ctr. of a rich silver ore, which was charged with five ctrs. of test lead, a silver button is obtained weighing 53·45 milligr. Since the difference between 50 and 60 is 10, and 53·45 would fall at about the third part of this difference, the loss for this proportion of silver would be $0{\cdot}32+\frac{0{\cdot}360{\cdot}\ 32}{3}=0{\cdot}32+0{\cdot}01=0{\cdot}33$ milligr., and the true amount of silver in the ore would be 53·45+ 0·33 53·78 per cent.

When the balance will only indicate 0·1 milligr. with difficulty, it is unnecessary to reckon the cupellation loss with more than one decimal figure, reckoning, however, as 0·1 each figure in the second decimal place which is greater than 5.*

Naturally the cupellation loss is only to be added for assays that do not serve as a control on commercial assays of ores; moreover, in blowpipe assays, where the silver must be measured on the scale, the loss is not reckoned in, because the proportion of silver is so trifling that the cupellation loss is often less than the error that may occur in the measurement itself.

Should the amount of silver in the substance to be assayed be so small that it comes among the lowest lines of the scale, it is safer for an unskilful person to weigh out several centners of the prepared assay powder, charge them each with the necessary amount of borax and test lead, fuse the prepared assay according to the method previously described, and then scorify the pieces of silver-lead so obtained by twos, or threes, at a time, down to small buttons. These small buttons, in which the amount of silver is already considerably concentrated, are then scorified all together on a fresh cupel, and the resulting button is cupelled fine.

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* Beginners, lacking the necessary practice, are apt to cupel too hot, and may still get too low a value, even after reckoning in the cupellation loss, as given in the tables. In order to learn by practice the temperature to be used in cupelling, particularly in the fine-cupellation, an accurately weighed button of pure silver is fused with five ctrs. of test lead, under a cover of borax-glass in the R. F., and after cupelling the silver-lead thus formed the pure silver button is weighed again. If the loss is greater than that given in the table for a button of this weight, it has been cupelled too hot, provided there has been no mechanical loss; if it is not greater, the proper heat was employed. Generally the greatest loss occurs in the fine-cupellation.

In this way the silver is united to one large button, the weight of which, as given on the scale, need only be divided by the number of centners weighed out to find the amount in one centner. It is assumed, however, in such a concentration assay, that the test lead is quite free from silver; otherwise the amount of silver in the quantity used must be determined by concentrating an equal amount, cupelling it, and deducting the silver found from the result previously obtained.

b. Assay of minerals containing compounds which cannot be decomposed by fusion with borax and test lead alone on coal.

Molybdenite is such a mineral, and an argentiferous specimen of this from the *Zwitter* of the Altenberg tin *stockwerk* in Saxony contained, according to Plattner, 0·176 °/₀ silver. This mineral is neither decomposed nor dissolved by borax, but is decomposed very easily with effervescence by soda, and regard must be had to this behaviour in the fusion of the assay. A small quantity of the molybdenite is first broken up as much as possible in the steel mortar, or between paper, if it cannot be pulverized in the agate mortar, and then 1 ctr. is weighed out and mixed with 1½ ctrs. soda,

1½ " borax-glass, and
5 " test lead.

This charge is wrapped up and fused like any other assay. The molybdenite is decomposed by the soda, its sulphur combining with the radical of the soda to form sulphide of sodium, while the liberated molybdenum partly combines with the test lead and partly volatilizes, coating the coal white. When the slag flows quietly, and there are no more scales of molybdenite to be seen, the lead button, which generally lies under the slag, is caused to come out by inclining the coal or crucible, and is treated with the O. F., until all the molybdenum, which forms an almost white and somewhat brittle alloy with the lead, is volatilized, and then the assay is allowed to cool. The addition of borax is necessary to prevent the sulphide of sodium from spreading over the coal. The lead is cupelled as before described.

c. Assay of metallurgical products which consist of metallic oxides and are easily reduced on coal.

Litharge and *abstrich* belong here. When resulting from the smelting of argentiferous lead ores, they always contain silver, even though in very small quantities, probably present as oxide. They are, however, generally so poor in silver that the amount cannot always be exactly determined from one blowpipe assay centner; but as they mostly consist of oxide of lead alone, which is very easily reduced, a large quantity can be taken, and the determination of the silver is free from difficulty.

Five ctrs. of each of these products is weighed out in the form of powder, mixed with an even spoonful of soda and the same amount of borax-glass, and the charge, wrapped in soda-paper, is treated in a cavity in good charcoal, or in a properly cut coal crucible, with the R. F., until all the oxide is reduced and the slag lies beside it, as a spherical bead, free from lead globules. Toward the end, however, the flame must be directed rather on the slag than on the reduced lead, otherwise a violent movement of the lead would ensue, with a possible loss of silver.

The lead reduced from litharge sometimes contains traces of copper, but is generally free from volatile metals, while that from abstrich often contains, besides a little copper, some antimony, arsenic, zinc, etc. These ingredients separate when, after ending the fusion, the slag alone, by the side of the lead, is treated with the R. F. The silver-lead is cupelled as in any other assay.

B. Metallic Compounds.

a. In which the silver is a chief ingredient.

Here are to be classed: *native silver, brightened, cement, refined, retort,* and *jeweller's silver,* and *silver coins.*

No regular fusion of the substances is necessary, but they must be melted together with test lead, in order to separate by cupellation, together with the lead, the easily oxidizable metals that are mixed with the silver. As these substances cannot be readily pulverized, it is unnecessary to weigh out exactly 100 milligr. for an assay, but a piece can be chiselled or broken off, which weighs from 80–100 milligr., but not more. If the surface is not clean, it must be made so by filing, before breaking it up.

The clean fragment is accurately weighed, placed in a hole made in charcoal with the borer, or in a coal crucible, and covered with one ctr. test lead and half a spoonful of borax-glass, if it is native silver, brightened, cement, or fine silver; but if it is a cupriferous retort or refined silver, or other silver alloyed with copper, it must be mixed, according to the proportion of copper, with two to five ctrs. lead and half a spoonful of borax-glass. The fusion is made with the R. F., and the test lead soon unites with the metallic compound and acquires a rotary motion. The union of the separate metals may be considered perfect when this has lasted a few moments; the blast is stopped, the whole allowed to cool, the fused assay raised from the coal, and the borax-glass separated as carefully as possible from the lead with a few strokes of the hammer. Although the lead can be easily combined with silver, copper, and many other metals by the blowpipe flame on coal without adding borax-glass, some of it readily oxidizes when it acquires a rotary motion, and the oxide formed being immediately reduced again on coming into contact with the coal, produces such a violent commotion in the fluid metals that spattering may easily occur. If, however, a little borax-glass is added and treated uninterruptedly with the R. F., the lead fuses easily with the metallic compound, and assumes the rotary motion without the occurrence of the evil mentioned.

The silver-lead is cupelled as before described. Compounds which have been fused with but one ctr. of lead can be subjected at once to fine-cupellation, but this is less to be recommended with a greater amount of lead, and at all events only for a practised person. In this case the oxidizing lead button, rich in silver, is caused to move away from the litharge to a free spot on the bone-ash, by slightly inclining the cupel, and is here cupelled to fine silver without letting it come in contact with the heaped-up litharge. Should the metallic compound contain several per cent. of copper, opportunity for oxidation must be afforded to this metal as much as possible during the scorification, so that in the fine-cupellation the rest of the copper may be separated by adding only one ctr. of test lead, according to p. 374.

When, therefore, a rich lead, containing a considerable quantity of copper, is to be treated, the oxidizing lead must not be surrounded by too much litharge in the scorification, but the cupel must be constantly held in a somewhat inclined position, so that the surface of the lead may be as free as possible and the copper may have sufficient opportunity to oxidize also. The silver button is removed from the cupel with the pliers, and then freed from the adherent fragments of the cupel by holding it firmly and pressing it upon the anvil, or it is held edgewise on the anvil with the small forceps and struck a few light blows with the hammer. As already remarked, one hundred milligr. of these metallic compounds will be rarely weighed out, and the silver obtained must be reckoned for one ctr., with due regard to the cupellation loss. When the compound contains gold, the amount of this is determined by the process given under the gold assay and deducted from the weight of the silver.

b. Assay of metallic compounds in which copper or nickel forms the chief, and silver only a minor ingredient.

Here are to be reckoned: *black copper, raw copper, refined copper;* also, *argentiferous copper coins,* under which head most of the small silver coins may be included (in Germany), *brass, German silver,* etc.

A small quantity must be prepared from a clean surface of the subtance, by beating it out and cutting it with the scissors, or by filing, so that an assay may be easily weighed out. In the case of black copper, raw copper, refined copper, argentiferous copper coins, and German silver, one ctr. is charged with twenty ctrs. test lead, or, to avoid so large a volume, one-half ctr. of the compound with

10 ctrs. test lead and
½ ctr., or one even spoonful, of borax-glass.

This is well mixed in the mixing capsule, wrapped in soda-paper, and fused in the R. F. as already described. The lead with the combined metallic compound must remain for some time in rotary motion, until no more of the metallic particles, which at first generally float upon the lead, can be seen. If the fusion were broken off earlier, those portions of the metallic compound not yet thoroughly combined with the lead would pass in part mechanically into the litharge during the subsequent cupellation. During the fusion any cobalt and iron, sometimes present in German silver, are oxidized and dissolved in the borax-glass, but the zinc, which is a chief ingredient of German silver, is volatilized. The lead after cooling is separated from the slag on the anvil and cupelled as before, but the button of silver obtained by the fine-cupellation generally spreads out, owing to the presence of copper, and it must be subjected to a second refining cupellation on the same cupel, with one-quarter to one-half ctr. of melted test lead, as directed on p. 374, so that the button may have a proper diameter for the determination of the silver on the scale. The weight of the silver button must necessarily be doubled if only one-half ctr. of the substance was taken for the assay; and in order to be safe with such an assay, two portions of one-half ctr. each may be weighed out, and the argentiferous lead buttons obtained by the scorification united in the fine-cupellation.

To assay brass for silver, 1 ctr. is charged with
10 ctrs. test lead and
1 heaped spoonful of borax-glass.

The charge, wrapped in soda-paper, is treated as before in the R. F., until the lead has united with the metal to be assayed, and has been in rotary motion with it for some time, while the borax-glass is free from lead globules; after which the flame is allowed to act only on the borax, and the zinc not yet volatilized in the fusion with the lead is thus completely removed. When the lead shows a clean surface, it is heated rather strongly for a few moments, and poured out on the anvil if the fusion was performed on an ordinary piece of coal, or allowed to cool slowly with the borax-glass if in a coal crucible. The cupriferous lead is cupelled just like that obtained from the foregoing metallic compounds rich in copper.

c. Assay of metallic compounds in which lead or bismuth is a chief ingredient.

Here belong the *argentiferous lead* and *bismuth*, obtained on a large scale; the mineral *chilenite* from Chili may also be mentioned here.

A small piece of the argentiferous lead is beaten out, cut up with the scissors, and from two to five, or even ten centners weighed out, according to the amount of silver expected. For the sake of dispatch a certain quantity is weighed out accurately, and then the amount of silver is calculated for one ctr. It is only advisable to begin at once with the cupellation in case of quite pure lead; generally it is better to fuse the weighed portion in an excavated coal or a coal crucible, and to treat it for a short time with the O. F. With lead containing much copper, regard must be had to the remarks on p. 374, about the complete separation of the copper in the fine-cupellation.

As bismuth is brittle, a quantity sufficient for several assays is broken off with the hammer on the anvil and made as fine as possible. Then a weighed portion, of about five ctrs., is fused in an excavated coal with borax-glass for some time in the O. F., and the fluid metallic button poured out upon the anvil, which will not cause the slightest loss if carefully done. The button is then subjected to the scorification, like argentiferous lead. In separating the oxide of bismuth thus formed from the argentiferous bismuth button, which is to be subjected to the fine-cupellation, great care must be taken to lose no fragments, since the bismuth is brittle. The button should never be lifted out of the oxide, but the latter must be gradually removed from it with the pliers. Argentiferous bismuth never yields a silver button with a bright surface, and it is therefore necessary to fuse the button obtained

from the scorification with a piece of test lead weighing thirty to forty milligr., and then proceed with the fine-cupellation.

d. Assay of substances in which tellurium, antimony, or zinc forms a chief ingredient.

Here belong *hessite* and *dyscrasite,* which occur in nature, and also *argentiferous antimony* and *zinc.*

One centner of the substance is fused on coal or in a coal crucible with five ctrs. of test lead, under borax-glass, with the R. F., and an opportunity is given to the metals in combination with the silver to volatilize by afterward treating the metallic button, rich in lead, with the O. F. alone. Zinc volatilizes rather easily; so does antimony, although it is somewhat difficult to remove the last portions; but tellurium can only be partially volatilized, and must therefore be separated through oxidation by cupelling it with a large amount of lead. When the antimony and zinc seem to be volatilized, the blast is stopped, the silver-lead separated from the slag, after the assay cools, and the cupellation is begun. When the lead is free from the volatile metals in question, it can be at once cupelled in two periods, but otherwise, as in case of native telluride of silver and other metallic compounds of silver containing tellurium, the scorification must be repeated, with fresh quantities of lead (five ctrs. each time), so long as the resulting silver button, containing lead, cools with a dark-coloured surface, before proceeding to the fine-cupellation. Finally, if after the fine-cupellation the silver button solidifies with a reticulated, dull, grayish-white surface, this indicates that traces of tellurium are still present. Another centner of test lead must be added and the fine-cupellation repeated. One centner of pure telluride of silver requires nearly twenty centners of test lead, and with about 62·7 °/₀ of silver suffers a cupellation loss of nearly 1·5 milligr. silver, so that only about 61°/₀ of silver can be actually obtained.

e. *Assay of metallic compounds in which tin forms a chief, or only a secondary ingredient.*

In this class belong *argentiferous tin*, *bell* and *gun metal*, and several other alloys containing tin, which are employed in the arts.

The substance is cut up, or finely divided in some way, and one centner weighed out and mixed in the mixing capsule with

5 to 15 ctrs. test lead, according to the amount of copper present,
50 milligr. soda, and
50 " borax-glass.

The mixture, wrapped in soda-paper, is treated on coal or in a coal crucible, with a strong R. F., until the tin or metallic compound is fused to a globule with the lead, and the soda, which prevents the easy oxidation of the tin, is melted to a glass with the borax. The metal alone is then touched with the blowpipe flame, for which purpose the blue flame is best suited, but only so that the very easily oxidizable tin may oxidize slowly, while the oxide is taken up by the fluid glass. When globules of reduced tin appear on the border of the slag, the blast is stopped and the assay allowed to cool, after which the lead, still containing tin, is treated on another coal with a spoonful of borax-glass in the O. F., in the same way as before with soda and borax, until the lead shows a bright surface, or one no longer covered with oxide of tin. It is then cupelled like any other argentiferous lead obtained from a fusion.

f. *Assay of metallic compounds in which mercury is the prevailing ingredient.*

This includes *native* and *artificial silver amalgam* and *argentiferous mercury*.

In weighing these substances, the scale-pans, if of silver or gilt, must, for well-known reasons, be covered with paper, and the balance then adjusted, after which one ctr. of the compound is weighed out and put into a small glass tube, sealed at one end and blown into a bulb, as shown in Fig. 65, p. 274. The tube is held somewhat inclined, and very gradually heated over a spirit-lamp, and the distillation continued until the metal remaining behind has been for some time at a red heat. By turning the tube and tapping on it, when it is somewhat cooled, all the separated mercury is collected into one drop and shaken out. When the metallic compound subjected to distillation was a silver amalgam, the silver remains in the bulb as a single porous globule, which can be easily shaken out, if the heat was not too high. This globule is fused with one, or if it appears to contain copper, with two or three ctrs. of test lead, under a cover of borax-glass in the R. F. on coal, and the resulting metallic compound, rich in lead, is cupelled in the usual way.

If the compound distilled was only an argentiferous mercury, a very trifling residue remains, which adheres firmly to the glass, and cannot be shaken out. The bulb of the tube must then be cut off, filled with a mixture of one ctr. of test lead and half a spoonful of soda, placed in an excavated coal, or a coal crucible, and the lead melted together with the residue of silver in a strong R. F. The resulting metallic compound flows out from the melting glass, and, after cooling, can easily be detached from the coal and glass, and cupelled. Several centners of mercury may be distilled, if the amount of silver is suspected to be very small.

g. Assay of metallic compounds in which iron or steel forms the chief ingredient.

Here belong, besides *iron* and *steel*, the iron *bears* which form, under certain circumstances, in smelting argentiferous ores and products.

Neither iron nor steel can be directly united with lead before the blowpipe, so they must first be combined with sulphur, and then they will yield up their silver to the lead just as readily as an argentiferous iron pyrites. Hardened steel must first be softened by heating, and then, after cleansing its surface from the resulting magnetic oxide by filing, the quantity necessary for an assay is reduced to a fine state by beating or filing. Iron *bears* being more or less brittle, can often be easily reduced under the hammer. From the divided metal, which may remain in pieces weighing from twenty to thirty milligr., one ctr. is weighed out, and mixed with

½ ctr. powdered sulphur,
8 " test lead, and
1 spoonful borax-glass.

The mixture is poured into a soda-paper cylinder and treated on coal, or in a coal crucible, with the R. F., until everything is fused to a fluid ball. The sulphur first combines with the easily fusible lead, and the iron, after continued blowing, begins to glow, taking a part of the sulphur from the lead to saturate itself, and combining then as proto-sulphide of iron with the lead, which still contains sulphur, to a quite fluid mass, which is surrounded by the melted borax. A single spoonful of borax-glass being insufficient to take up all the iron which must be oxidized by treating the assay with the O. F. after this fusion, another heaped spoonful of the flux is then melted together with the already fused globule, and the whole treated with a powerfully oxidizing flame, until the impure lead begins to separate from the glass. The coal is now held so that, for the most part, only the lead is touched by the outer flame, in order that the sulphur may pass off and the iron be oxidized and dissolved in the borax. After all the sulphur is gone and the iron separated, the blast is stopped, and the silver-lead, which has a bright surface, is allowed to cool. If it is of a white colour, it is cupelled as usual, and the weight of the silver button ascertained; but if it is black and brittle, it must be exposed to a second oxidation before it can be cupelled.

2. THE GOLD ASSAY.

Like silver, gold can be separated from its combinations in the dry way, and it is therefore possible to ascertain with the blowpipe the amount of gold present in ores, minerals, and metallurgical and artificial products. In this method gold suffers no cupellation loss, since it cannot be oxidized.

Gold, however, seldom occurs in nature without containing some silver, while silver ores are very frequently more or less auriferous, and since the silver cannot be separated from the gold in the dry way, the separation of gold in a pure state is somewhat more complicated than that of silver.

With reference to the quantitative assay for gold, the various minerals, ores, and products may be divided into:

A. Gold ores, auriferous silver ores, and *argentiferous* and *auriferous metallurgical products.*

B. Metallic compounds, viz.,

- *a.* Such as consist only of gold and silver;
- *b.* Such as contain, besides gold and silver, other metals, as copper, platinum, iridium, palladium, and rhodium;
- *c.* Such as consist of gold and mercury.

A. Assay of Gold Ores, Auriferous Silver Ores, and Auriferous and Argentiferous Metallurgical Products.

This includes: 1. *Native tellurium*, which is said to contain 0·25 to 2·78 % gold, but no silver. 2. All the minerals which contain silver as well as gold. 3. The *auriferous iron* and *copper pyrites* occurring in various places 4. *Rohstein* and *lead matt* obtained from auriferous silver ores. 5. The grindings and gold scraps of gold and silver smiths.

In the case of gold ores proper, containing little or no silver, the quantity of assay powder necessary for several assays is prepared, and an assay made from it just as in the silver assay. After the cupellation, the colour of the button will show whether the gold is pure or contains silver, since even two per cent. of silver is sufficient to give the gold a brass-yellow colour. If the button has a pure gold colour, its weight may be immediately determined on the balance, or on the scale; but a lighter colour indicates silver, and then a further separation must be effected, as will be described hereafter.

From the other substances, containing more silver than gold, a quantity of assay powder, sufficient for ten or fifteen assays, is prepared, and a preliminary assay of this is first made for silver. From the amount obtained a calculation is made of the number of assays necessary to obtain a quantity of silver so great that the gold in it may be quantitatively determined. If it is a substance containing only about 0·11 pound of silver in a hundredweight, or 0·11 °/₀, and in which but little gold is suspected, more assay powder must be prepared, and at least twenty-four assays weighed out; but if it contains more silver, perhaps as high as 0·28 °/₀, ten or fifteen assays will be enough. It is in general advisable to make as many assays as possible, with a substance poor in silver, because the proportion of gold to silver can be determined quantitatively only in a portion of the alloy which is large enough to be weighed on the balance. If the substance contains several per cent. of silver, only a threefold or fivefold assay is made.

The separate assays are charged precisely as in a silver assay, with borax and test lead; if the substance contains copper, the quantity of lead must be increased in proportion to the probable amount of that metal present. Each assay is fused exactly according to the directions given for the silver assay; the fine-cupellation of the raw lead from each assay, however, is not performed separately, but in the manner now to be described.

When all the assays have been fused, and the lead freed from slag, and hammered into cubes, these are placed two or three at a time, if their united weight does not exceed fifteen ctrs., on a well-heated cupel, and the scorification begun as described in the silver assay. After the scorification has been carried as far as is there stated, it is stopped, and the litharge and lead button lifted off from the bone-ash, which is not yet permeated with oxide, and set aside, while a new cupel is made, heated well, and another scorification performed with two or three more pieces of raw lead. This operation is performed on all the lead, and the resulting buttons, which contain the concentrated silver and gold, are all placed on a fresh, well-heated cupel, and the concentration of the gold and silver continued until the rich lead is only the size of a large mustard-seed. This button is then subjected to a fine-cupellation on another cupel. When the substance contains a great deal of copper, so that the silver is not pure after the brightening, a little test lead is added and the button cupelled fine on a free spot of the cupel. The auriferous silver button obtained is accurately weighed, and then parted in the manner described below, under metallic compounds. For the parting, the auriferous silver button must always be large enough to weigh, since its true weight cannot be read upon the scale on account of the greater specific gravity of the gold.

When pure pyrites, or very pyritiferous ores containing silver, or metallurgical products consisting chiefly of metallic sulphides, are to be assayed for gold, another method must be employed to concentrate the silver and gold contained in them. Should the finely-powdered ore or product contain not less than about 0·11 $^{0}/_{0}$ of silver, twenty-four to thirty-six ctrs., according to the richness, are weighed out in portions of three ctrs. each, and the separate portions put into a clay capsule which has been painted with reddle, and roasted without the addition of any carboniferous substance, as in the quantitative copper assay. When no more fumes of sulphurous acid can be smelled, the roasted stuff is rubbed in a mortar and again ignited in the capsule, until no signs of any escaping sulphurous acid can be noticed. This is done with all the weighed portions. The roasting can be performed in a very short time if one is provided with a gas-lamp, or a spirit-lamp with double draught,—as it is then only necessary to heat the whole weighed quantity of ore at once to a low red heat, in a thin, flat porcelain dish, over the lamp, with access of air, stirring it from time to time with the iron spatula, and keeping it in a glowing state, until the smell of sulphurous acid is no longer perceptible.

After the whole twenty-four or thirty-six ctrs. are roasted, the ore, or product, is put in a porcelain vessel, corresponding to the quantity of the substance and enough hydrochloric acid added to dissolve the oxides mentioned below. The porcelain vessel, covered with a watch-glass, is set upon a wire frame stretched over a brass ring, above the feeble lamp-flame, and the solution is begun with the aid of heat. Sesquioxide of iron, oxide and sulphate of copper, and the simultaneously forming chloride of silver, are dissolved. The gold remains in the metallic state if the roasted ore is free from metallic oxides which dissolve in hydrochloric acid with evolution of chlorine, but if it contains such oxides, for instance, proto-sesquioxide of manganese, the gold also goes into solution. Any earthy admixtures not soluble in hydrochloric acid likewise remain behind. After complete solution of the soluble portions, the whole is evaporated to dryness, preferably on the water-bath, and the remaining mass is warmed with enough water to separate the soluble chlorides from the now insoluble chloride of silver and the other insoluble portions. The whole is then treated with a solution of sulphate of iron, to precipitate the gold in solution, stirred, and allowed to settle. When the liquid is clear, it is filtered, the precipitate and residue washed with water, and dried on the filter in a porcelain dish over the lamp-flame, without unfolding it. The dry filter is then unfolded, five ctrs. test lead and a spoonful of borax-glass carefully mixed with the powder upon it, and the filter folded again. After which, the upper part is cut off, if none of the charge has touched it, while the lower part with the charge is wrapped tightly together and placed in an excavated coal, or a coal crucible. The paper is first charred and mostly destroyed by a gentle O. F., and the whole is then fused with a good R. F. The test lead decomposes the chloride of silver, and the silver and gold unite with the lead, while any earthy matters present are dissolved by the borax. The raw lead is then cupelled in two operations, as in the silver assay, and the auriferous silver button further treated, as will be hereafter described in the parting of gold and silver.

If pyrites or matt-like products containing still less than 0·11 $^{\circ}/_{0}$ of silver are to be assayed for gold, a larger quantity must be taken, and the above method would then be rather tedious. The use of other apparatus and contrivances is recommended in such cases, the blowpipe being also used with advantage here, as will be seen from the following description :—

In one method fifty to two hundred grms. of the finely-powdered substance are weighed out on a less delicate balance, and roasted on a piece of sheet-iron, which has its edges turned up and has been brushed over with a few coats of fire-clay and water, and then dried. This is heated over a charcoal or coke fire in a small portable furnace, and under a chimney with a good draught.* The roasting substance is stirred with an iron spatula, until, with a rather strong red heat, no more sulphurous acid can be smelled, and when cool it is rubbed in an iron mortar, after which it is again ignited on the sheet-iron, until not the least smell of sulphurous acid can be perceived. After cooling it is moistened with water in a porcelain dish, but just enough to leave it in a still loose, porous state, in which it can be best treated with chlorine. If the roasting was thorough, the gold, which is present in the metallic state, can be converted into chloride and extracted as such by fresh chlorine water, free from hydrochloric acid; but if small quantities of metallic sulphides and arsenides are still present, the chlorine is best used in the gaseous state, observing the following conditions, which secure the perfect extraction of the gold:

1. The substance to be treated must be as free as possible from metallic iron, as, for instance, fragments of stamp-heads, which can only be determined with a magnet; any fragments found must be removed.

2. The careful roasting of the finely-divided substance is presupposed, so that there may be as few undecomposed metallic sulphides and arsenides as posssible.

3. The cholrine gas introduced into the moist roasted sub-

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* In lack of a suitable portable furnace, a large graphite crucible, about 0 3 met. high, such as is used for fusing metals, can be employed; in the side of this, near the bottom, a hole about seven centim. square is made, with several small holes around the upper edge of the crucible, inside of which a suitable iron grate is fixed.

stance must be free from hydrochloric acid, which not only easily dissolves all metallic oxides present, but also, in presence of sulphide of iron, etc., evolves sulphuretted hydrogen, and this both decomposes the chloride of gold already formed, precipitating the gold as sulphide, and also absorbs the chlorine itself, so that but little gold can be extracted. The gas is therefore first freed from all hydrochloric acid gas, and the extraction of the gold is performed in the following way:

In the glass cylinder *A*, Fig. 72, which is about two hundred to two hundred and fifty millim. high and sixty millim. wide, with a neck eighteen to twenty millim. wide on the side near the bottom, is placed a layer of small pieces of quartz, reaching above the neck; above this is strewn a layer of coarse quartz sand, and then a layer of fine sand, forming a filter. The moist roasted ore is laid as loosely as possible on this, and the top of the cylinder closed with a caoutchouc cap, in which is fixed a tube that dips into the cylinder *B*, where there are shavings or a roll of stiff blotting-paper, to be subsequently moistened with alcohol. The chlorine necessary for two hundred grms. of roasted ore is then produced from ten grms. powdered binoxide of manganese, forty grms. ordinary hydrochloric acid, and ten grms. sulphuric acid, which has been previously diluted with an equal quantity of water. These are thoroughly mixed by agitating the flask in which

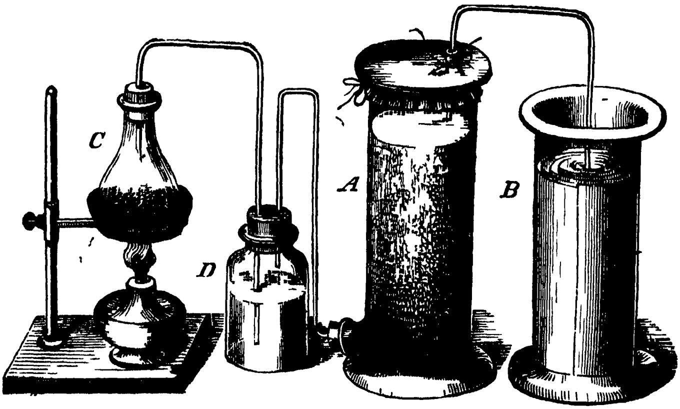

FIG. 72.

they are put, and this is heated on a sandbath over a spirit-lamp, being connected by a glass tube with the wash-bottle *D*. This bottle has a wide neck, closed with a cork, and is more than half full of pure water. After yielding its hydrochloric acid gas to this water the chlorine passes on into the cylinder *A*, as shown in the figure, and thus, if the apparatus is air-tight, to the very end of the tube which opens near the bottom of *B*, the extraction may be performed in the laboratory without any odour of chlorine becoming perceptible, since chloral and hydrochloric acid, both inoffensive, are formed from the combination of the superfluous chlorine with the alcohol.

The flask is at first very gently heated to avoid too rapid evolution of gas, but afterward somewhat stronger. The absorption of the gas in the cylinder *A*, and the change of colour in substances containing a notable amount of gold, can be very plainly seen, and also the appearance of the yellowish gas in the empty part of the cylinder, as well as its final flow into *B*. The fewer the undecomposed metallic sulphides and arsenides in the roasted substance, so much the sooner does the chlorine appear above it, while with a more considerable amount it makes its appearance later, and a decided evolution of heat is noticed in the cylinder. All the finely-divided metallic gold present is changed into chloride as soon as the chlorine appears in the empty part of the cylinder, but this is by no means the case with the gold which is contained in the parts not decomposed by the roasting. The gas must pass through the powder for at least an hour before the extraction can be regarded as complete and the apparatus taken apart, in which operation the cork should first be removed from *C*, to prevent the reflux of water when the lamp is taken away.

The cylinder *A* is then placed on a stand and the small neck closed with a cork, in which is a short, narrow tube, bent downward. By carefully pouring on small quantities of hot water, the chloride of gold, and any other resulting soluble chlorides, are then washed into a beaker, the cylinder being inclined toward the end of the operation, so as to leave nothing in the bottom. If the neck is too high above the bottom of the cylinder, the space can be filled up with melted pitch before laying in the quartz. After adding hydrochloric acid, and then enough solution of sulphate of iron to precipitate the gold, the liquid is strongly stirred with a glass rod and left until all the gold has settled, which occurs in a short time if the liquid is kept warm. The separated gold is collected on a filter, washed, and the filter dried in a porcelain dish over the lamp, after which it is ignited with access of air in a platinum capsule. The residue is mixed in the capsule with one to two ctrs. test lead and a little borax-glass, wrapped in soda-paper and fused like a quantitative silver assay. The weight of the button obtained by cupelling the auriferous lead is then determined on the scale or the balance. If 200 grm. =2,000 blowpipe ctrs., of raw pyritiferous ore were used, the weight of the gold button is divided by 2,000 to find the amount of gold in 1 ctr. of the ore.

The gold thus obtained is generally free from silver, because the chloride of silver formed is insoluble in pure water; if, however, the substance was imperfectly roasted, so that a considerable quantity of other chlorides could form, the water at first used in washing the substance after treatment with chlorine forms a concentrated solution and takes up some chloride of silver, which, although the solution is diluted more and more with water, only separates very slowly. In this case the gold may contain a little silver, and must be separated as will be described under alloys.

Another method, feasible for any assayer who has a muffle and wind-furnace at hand and is practised in the use of the blowpipe, is the following. A very poor ore may be used, which would necessitate the treatment of three or four pounds in order to obtain a weighable button of gold by the ordinary muffle assay. From seventy-five to one hundred and twenty grms. of the ore or product in question are first separated by any of the methods known to all assayers, viz., by a scorification assay in the muffle, followed by cupellation of the raw lead in the cupel, or by smelting the roasted substance with alkaline fluxes, litharge, or test lead, and other reagents, in clay crucibles, concentrating the lead by scorification under the muffle and then cupelling the enriched lead. The auriferous silver button thus obtained is weighed with the ordinary assay weights, the amount of silver contained in the test lead or litharge employed is deducted, and from the result the amount of silver and gold together in the ore is calculated. Then, after separating the silver by the method to be directly given, the weight of the gold is determined on the measuring scale, and from it the proportion of gold and silver separately ascertained. If the system of gramme weights is used throughout, there will be no difficulty whatever in making the necessary calculations.

B. Metallic Compounds.

a. Consisting only of gold and silver.

This includes *native gold, gold alloyed* with *silver,* and the *argentiferous gold* or *auriferous silver* from the assay of auriferous minerals, ores, and products.

There is no sure way of easily dissolving or slagging off one of these metals in the dry way, so as to separate the other pure and without loss, and it is necessary to perform the separation with nitric acid, both in the commercial and blowpipe assays. In the separating process, which is called *parting,* the proportion of silver to gold in the alloy must be not less than 2·5 : 1; otherwise the silver will be imperfectly, or not at all dissolved. The amount of gold must, therefore, be previously ascertained, and if it exceeds this limit the lack of silver must be supplied. *Touch needles* are used in the muffle assay, and in blowpipe assays, where very small quantities are employed; although not indispensable, these may be replaced by a small stock of alloys of known composition, which with a touchstone are of great advantage when frequent gold assays are made. Gold containing only 2 °/₀ of silver has a light brass-yellow colour, and if it has 60 °/₀ no yellow colour is perceptible. From its more or less yellow colour the approximate composition of the alloy can be estimated, as well as the amount of silver necessary to produce the required proportions, but this may be more exactly ascertained from the streak on the touchstone.

When native gold, of a brass-yellow colour, is to be assayed for fine gold, it may be assumed that the amount of silver is unimportant, and fifty to eighty milligr. being weighed out, are fused with two and a half times the quantity of silver free from gold, which has been reduced from chloride of silver with a little borax-glass on coal in the R. F. A very light brass-yellow indicates a more considerable proportion of silver, and in this case only twice the weight of silver is added. When the alloy is silver-white, consisting perhaps of 60 °/$_0$ silver and 40 °/$_0$ gold, the proportion of silver cannot be estimated, and it is then necessary to fuse the weighed portion with fully half as much pure silver.

Care must be taken in fusing the alloy with the silver and borax that the gold is equally distributed throughout the silver, and on this account the button must be kept for some time in a fluid state, while the R. F. is allowed to act only on the borax bead. The alloy obtained from minerals or gold ores proper is generally richer in gold than in silver, and hence in this case also the button should be fused with two and a half times its weight of pure silver. As regards the alloy obtained from auriferous silver ores, pyrites, or matt-like products, the gold is generally much less than one-third or one-fourth, and no silver need be added to such a compound.

When the silver in native gold, or in an artificial alloy of gold and silver, is also to be determined, the weighed metallic compound, before being fused with the pure silver, must be cupelled with one or two ctrs. of test lead, to remove any admixture of easily oxidizable metals, as iron, copper, etc., and the resulting button weighed; after which the silver is determined by difference when the weight of the separated gold has been ascertained. When regard must be had to the cupellation loss suffered by the silver, all of the litharge and the bone-ash which is permeated by it must be reduced on coal, with the addition of soda and borax-glass, the resulting lead cupelled and the weight of the silver button as ascertained on the scale added to the amount of silver previously found by difference.

The surface of the alloy to be parted is increased by hammering it out between paper, and it is then heated to redness on coal with a weak blowpipe flame, so as to lessen the increased density, after which it is bent into a small roll. Very small buttons do not require this preparation. It is now laid in a small porcelain vessel, and chemically pure, moderately strong nitric acid poured over it in slight excess. The vessel is then placed over the free lamp-flame on the wire frame, and covered with a watch-glass. The silver dissolves readily, leaving the gold in a black metallic mass, which preserves the shape of the compound when considerable gold is present, but otherwise is divided into several portions. As soon as the yellow fumes cease, the flame is brought nearer, and the acid heated to gentle boiling for a few minutes, and then the wire frame is turned to one side and the vessel allowed to cool until it can be held with the fingers. Then the silver solution is carefully decanted from the gold on the bottom with the aid of a glass rod, and, for the sake of certainly removing all the silver from an alloy rich in gold, the remaining gold is boiled with fresh acid. When all of the silver solution is decanted, the vessel is half filled with distilled water and again boiled, after which this water is decanted and the washing repeated several times, especially if the amount of gold is considerable.* When the last wash-water has been poured off, the gold is dried thoroughly in the vessel over the flame, care being taken not to heat it too strongly, or it will adhere very firmly to the porcelain. The dried gold is generally in a finely-divided state when its amount is trifling, and it is then advisable to mix it in the vessel with a little borax-glass and about one ctr. of test lead; pour this mixture into the mixing capsule, and then transfer it to a soda-paper cylinder, and fuse the whole in a moderate R. F. on coal. After thorough fusion, the auriferous lead is separated from the glass by very carefully hammering it between paper, and is cupelled fine at once on a well-heated cupel of sifted bone-ash, covered with elutriated bone-ash. Gold forms a brittle alloy with lead, if in considerable proportion, and hence loss may ensue if the rich lead is strongly hammered or pressed. The gold button is weighed or measured, according to its size.

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* The silver solution is decomposed by adding hydrochloric acid, and the resulting chloride is washed, dried, and preserved until a quantity is on hand, when it is reduced by placing it in a porcelain vessel, pouring water containing some hydrochloric acid over it, and then adding a piece of zinc. The well-washed reduced silver is melted to a button in the R. F. on coal, with a little borax.

Should the amount of gold be considerable, it may be shaken into a platinum spoon or a clay crucible and heated to redness. The spoon is heated over the spirit-lamp, the clay crucible in a square coal, as described in the quantitative lead assay, the crucible and coal being uncovered and only a low red heat applied. The heated gold, which has baked together and has a light gold-colour, is then weighed.

When the gold has been extracted from an ore by chlorine, the silver remains in the ore as chloride, and its amount must be ascertained by making a duplicate blowpipe assay for auriferous silver.

If the gold button is too small to be weighed, it must be measured on the ivory scale, (p. 27) as in the case of the silver buttons. Those gold buttons that are larger than the transverse No. 26, are more accurately determined by being weighed on the balance. The following is the gold scale and corresponding percentage:—

No. of the Transverse Line.	Percentage of Gold.	No. of the Transverse Line.	Percentage of Gold.
26	1·06523	13	0·13296
25	0·94609	12	0·10473
24	0·83784	11	0·08066
23	0·73741	10	0·06061
22	0·64534	9	0·04418
21	0·56134	8	0·03103
20	0·48485	7	0·02079
19	0·41570	6	0·01309
18	0·35346	5	0 00757
17	0·29776	4	0·00388
16	0·24824	3	0·01164
15	0·20455	2	0·00048
14	0·16630	1	0·00006

b. Assay of metallic compounds which contain other metals besides gold and silver, as copper, platinum, iridium, palladium, or rhodium.

1. *Gold* alloyed with both *copper* and *silver*.

Thirty to fifty milligr. of the alloy are weighed and fused with 3·5 to 8 ctrs. test lead, according to the copper present, with a good R. F. under borax-glass on coal, and the resulting lead cupelled like any other lead containing copper (p. 374). Should the argentiferous gold button resulting from the fine-cupellation seem too impure, owing to a small admixture of copper, it must be cupelled fine at once on a free spot of the cupel after adding one ctr. of test lead. When both the gold and silver are to be determined, the refined button is weighed and the two metals are then parted as above described. There is undoubtedly some cupellation loss in cupelling such an alloy, but this can be ascertained by reducing the litharge and treating it as described on p. 399.

Suppose that our alloy weighed 40 milligr. and we obtained a gold and silver button of 28 milligr. which yielded 20 milligr. of gold after parting. Then since pure gold is 24 carats fine, we would have 7·2 carats of copper,

40 : 24 :: 8 : 4·8 ,, silver,
40 : 24 :: 20 : 12·0 ,, gold.

24·0 carats.

2. Here are included alloys of gold with platinum, with platinum and silver, and with platinum, silver, and copper.

a. Gold and *platinum*.—When the amount of platinum is considerable, the separation is best effected by dissolving thirty to fifty milligr. of the alloy in aqua regia (three parts of hydrochloric and one of nitric acid), by warming it in a beaker-glass, adding chloride of ammonium to the solution, and evaporating the whole to dryness in a porcelain dish at a moderate temperature, so as not to decompose the salt. The residue is then washed on a small filter with alcohol of 75° to 80°, until a fresh portion of alcohol is no longer coloured yellow. By this means the gold is dissolved out and may be precipitated in the metallic state, after adding water to the solution and removing the alcohol through evaporation, by a solution of sulphate of iron, or by warming it with a solution of terchloride of antimony, allowing it to settle and collecting it on a filter. The filter is unfolded, dried in a porcelain dish over the lamp, folded up again, and incinerated over the agate mortar, being held up by one edge with the forceps. The ashes are mixed with about fifty milligr. borax-glass, wrapped in soda-paper, and the divided gold fused to a button with the O. F. on coal, while the ashes are slagged off. This button is freed from the borax between paper on the anvil and weighed.

As alloys containing much platinum rarely require to be treated, this separation, which requires much time, is seldom made. It is more frequently the case that gold containing but a small quantity of platinum is to be assayed for fine gold. Thirty to fifty milligr. of such an alloy are fused thoroughly with three times as much pure silver, the alloy then hammered as thin as possible, heated to redness, rolled up and treated a few times with nitric acid. By boiling the alloy somewhat longer than if it were free from platinum, the latter is dissolved with the silver, leaving either pure gold, or, if there was nearly ten per cent. of platinum in the original alloy, gold with a little platinum. In the first case the gold is boiled with distilled water, washed a few times with cold water, dried, heated to redness in a platinum spoon, and weighed. In the second case, however, the gold does not show a pure gold colour after being weighed and fused with a little borax on coal, and it must be again fused with three times its weight of silver, after which the platinum can be completely removed by nitric acid.

β. *Gold, platinum,* and *silver.*—When no regard is had to the silver, the method just described is exactly followed, but if the silver is to be determined, it must first be extracted with sulphuric acid. To do this with proper accuracy the alloy should contain, according to Chaudet, for one part of gold and platinum not less than one and a quarter nor more than two parts of silver, because some platinum seems to dissolve with more silver. When silver is lacking, an accurately weighed quantity of pure silver must be added, and if gold is lacking, the alloy must be melted with pure gold, to secure the necessary proportions of the metals. Thirty to fifty milligr. of the alloy being weighed out and brought to the proper proportions by fusing it with gold or silver and borax-glass on coal, the button is beaten as thin as possible, heated to redness, and rolled up. After being weighed to see that no mechanical loss has occurred, it is covered with concentrated sulphuric acid in a porcelain vessel and boiled for ten minutes. After cooling somewhat, the acid solution, containing sulphate of silver, is decanted and the porous metallic residue boiled five minutes longer with fresh acid to complete the separation of the silver. The remaining roll is boiled with distilled water, dried, ignited, and weighed: the difference gives the weight of the silver.

The gold and platinum are then separated by fusion with silver and solution in nitric acid, as described under *a*.

γ. *Gold, platinum, silver,* and *copper.*—The copper is first separated by cupelling the alloy with test lead as described on p. 402. If the gold cannot be made fine, owing to the presence of too much platinum, the rest of the lead is separated by means of boracic acid, as given in the qualitative assay, p. 286; it being assumed that the copper has been separated on the cupel. The silver is then removed with sulphuric acid, and the gold and platinum separated as above.

3. *Gold containing iridium.*—The iridium can be very easily detected and separated by treating the alloy with aqua regia, which dissolves the gold and leaves the iridium as a black powder. When the decomposition is effected, the gold solution is diluted with water, filtered, and the iridium well washed. The gold is precipitated either with sulphate of iron or by warming it with terchloride of antimony, according to p. 402, and determining it as there directed. Any copper in the alloy is first removed by cupelling it with three to five parts of test lead, and subsequently every trace of lead is removed with boracic acid on coal, if the gold containing iridium cannot be made fine by simple cupellation.

4. *Gold containing palladium.*—Thirty to fifty milligr. of the gold containing palladium are fused with three times as much silver and treated with nitric acid, as described in the separation of gold from platinum. The gold remaining behind is boiled and washed with distilled water, ignited, and weighed.

Should the alloy not be free from easily-oxidizable metals, the separation of the gold from the palladium is preceded by cupellation with three to five parts of test lead, and, if necessary, treated with boracic acid on coal. The silver is precipitated from the diluted solution by salt and the palladium in the metallic state by zinc.

5. *Gold containing rhodium.*—According to del Rio, when the gold is the prevailing constituent the alloy is dissolved without residue in aqua regia, and the gold can then be precipitated, free from rhodium, by sulphate of iron. According to Berzelius, rhodium can be separated from platinum, iridium, and osmium, by fusing the finely-divided alloy with bisulphate of potassa, which dissolves it and leaves the other metals. Gold being likewise insoluble, this method can be applied to gold containing rhodium if the alloy is converted into a thoroughly porous state. Thirty to fifty milligr. of the alloy are weighed out, fused with borax-glass and three times the weight of pure silver, then beaten out as thin as possible, annealed and bent into a roll. This is treated with nitric acid until all the silver is dissolved, leaving the spongy gold and rhodium in the shape of the roll. The silver solution is decanted, the roll boiled and washed well with water, then dried in the porcelain vessel. It is now gradually heated in the large platinum spoon or a platinum dish with a sufficient amount of bisulphate of potassa, until the salt is fluid at a low red heat. The rhodium and any remaining silver dissolve with lively evolution of sulphurous acid, imparting a dark red almost black, colour to the salt. When the evolution of sulphurous acid ceases and the fluid salt becomes quiet, it is poured off from the gold upon the iron anvil as completely as possible, using the iron spatula for the purpose, and the fusion is repeated with fresh bisulphate. This portion, which is only slightly coloured, having been poured off, the gold is boiled a few times with distilled water in a porcelain vessel, dried, ignited, and weighed. It is safe to ascertain, in case much rhodium was present, whether this gold is quite free from rhodium, by again fusing it with three times its weight of silver, dissolving it in the shape of a roll in nitric acid, treating it again with bisulphate of potassa, washing, drying, igniting, and reweighing it. A trace of remaining rhodium will be

shown by the pale yellowish colour imparted to the bisulphate, and if weighable is also detected by the difference in the weight of the gold.

c. Assay of metallic compounds of gold and mercury.

Gold amalgam occurs in nature, and is also artificially produced by the amalgamation of gold ores and for gilding metallic instruments, etc.

About fifty milligr. of amalgam are weighed out, the scale-pans being covered with paper if of silver or gilded, and then distilled just like the silver amalgam, p. 387. The remaining gold is cupelled with one ctr. test lead and weighed. If a light colour of the button indicates silver, this is to be separated by the process described on p. 400, and the amount of gold and silver reckoned, after again weighing the gold button obtained from the separation.

When one ctr. of the mercury to be treated contains a weighable quantity of both gold and silver, the method given on p. 388 is followed exactly, and the argentiferous button melted with two to three parts of pure silver and separated as above. The difference in weight between the argentiferous gold button and the gold with sulph[illegible] gives the weight of silver. When the mercury is very poor, and one ctr. will not afford a weighable argentiferous gold button, several centners are distilled in a small glass retort with a receiver over the spirit-lamp, or the distillation may be made in a tube blown out at one end, which is charged with one ctr. of amalgam, the condensed mercury cleaned out, and another centner of amalgram charged and distilled, continuing this until a weighable crust of gold and silver is observed in the bulb. The operation is then continued as directed for mercury containing silver (p. 388). The silver and gold are separated and determined as before.

3. THE ASSAY FOR COPPER.

Copper can be separated with little difficulty by means of the blowpipe, in the metallic state, when in combination, even if combined with other metals. Regard must, however, be had to the different ways in which it occurs, both in nature and in artificial products, because its quantitative determination varies in the different cases. Substances containing copper are therefore most suitably classified as follows:

A. Ores, minerals, and *metallurgical products* :—
  a. Containing volatile constituents;
  b. Containing copper in an oxidized state, free from, or combined with, acids and water; or slagged with earthy matters, or combined in any other way.
B. Alloys in which copper is the prevailing, or an accessory, ingredient :—
  a. Plumbiferous copper and cupriferous lead;
  b. Compounds of copper with iron, nickel, cobalt, zinc, and bismuth; either singly, or several at the same time, while lead, antimony, and arsenic frequently accompany it;
  c. Copper containing antimony;
  d. Copper containing tin.

Before the fusion of substances belonging to class *A, a,* preliminary roasting is necessary to remove the sulphur and arsenic.

A. Assay of Ores, Minerals and Products,

which,—*a, contain volatile constituents,* as *sulphur, selenium,* and *arsenic.*

This includes *copper ores* dressed on the large scale; among minerals, the compounds of copper with *selenium, arsenic,* and *sulphur*; among metallurgical products, *copper, matt, regulus, cupriferous Rohstein, lead matt, tutty,* or *cadmia,* etc.

ROASTING THE ASSAY.

One ctr. of the assay powder is mixed in an agate mortar with three times its volume of pure dry charcoal-dust, or with twenty to thirty milligr. of graphite, which is preferable in nearly all cases, and especially for substances very rich in arsenic. Minerals which, on account of a considerable proportion of sulphide of antimony or bismuth, fuse easily together at a low red heat, viz. *chalcostibite, bournonite, emplectite,* receive an addition of fifty milligr. pure sesquioxide of iron, or pulverized hematite, which prevents them from fusing together, and has not the least injurious effect on the subsequent separation of the copper. The carefully-mixed charge is transferred to a clay capsule, which has been painted with reddle, and is then spread out with the iron spatula.

A square coal is then fixed in the coal-holder and so much of the coal cut out on one side of the cavity with the knife as is indicated by the slit *b*, Fig. 48, p. 36, so that the space cut out forms a channel leading to the cavity. The wire and platinum shield (p. 37) are fixed in the cavity, and then the capsule is set upon the wire with the forceps, as shown in Fig. 73. In all these assays the aperture in the blowpipe tip should not be too narrow. At first a moderate O. F. is directed through the lower part of the channel *a* into the empty space below the capsule, so as to bring the sides of the cavity and the capsule itself to a low red heat. The point of the blue flame must reach little or not at all within the coal-holder. When the substance begins to glow, if *coal-dust* has been used, a moderate blast must be continued for some time, so that the particles of ore may not bake together, much less *sinter* or fuse, while the coal is consuming. When this has consumed, which can be ascertained by examination with the iron spatula, first warming it in the spirit-flame to prevent adhesion of the ore, the capsule is removed, and the somewhat cool charge poured into the agate mortar and rubbed fine. The ore will usually have changed in colour, and lies in a quite porous state in the capsule, so that it is seldom necessary to detach still adhering particles with the spatula.

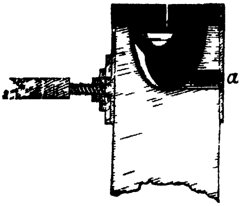

Fig 73

This roasting is completed in about ten minutes, and most of the volatile substances,—sulphur, selenium, arsenic, and part of the antimony, are removed, while the addition of coal has prevented to a great degree the formation of sulphates and arsenates. Some arsenic and sulphur remain, however, partly as unaltered sulphides and arsenides, and partly as acids combined with the metallic oxides formed, and to remove them as completely as possible a second roasting is required. The substance is therefore again mixed in the mortar with three times its volume of charcoal-dust, the capsule painted anew with reddle, and replaced with the charge upon the wire, when the roasting is continued. As soon as the intermingled coal is in full glow, a somewhat stronger heat is employed, and the presence of still volatilizing constituents tested by smelling. When the charge ceases to give any odour, the rest of the coal is burned off and the assay regarded as thoroughly roasted. If, however, fumes are seen to escape, the assay must be again rubbed in the mortar, coal-dust added, and a third roasting undertaken, during which the presence of volatile constituents is again tested by smelling. When no more fumes can be smelled and the coal-dust is consumed, the capsule is removed and allowed to cool. Should fumes still be observed, which is only the case with substances containing much arsenide of nickel, even a fourth roasting is necessary; but generally the second is enough. Since it would be tedious to reweigh the assay, and thus ascertain whether its weight remained constant, the absence of any odour of volatile bodies from the glowing assay is regarded as indicating the completion of the roasting when coal-dust is added. The colour of the roasted substance affords a tolerably sure sign of its richness in copper. The darker its blackish-brown colour after cooling, so much the richer is it in copper, while a redder or lighter shade indicates less of this metal.

In roasting with *graphite*, the assay is kept at a red heat from the beginning of the roasting until no more volatile substances can be smelled. The graphite is less easily destroyed than the charcoal-dust, and being, therefore, longer in direct contact with the substances, constantly exerts a deoxidizing influence on the volatile constituents, preventing the formation of sulphates or arsenates better than the coal, which has to be added anew in order to decompose them. When, therefore, the fumes cease, the capsule is removed, and the substance, mixed with still undestroyed graphite, is carefully rubbed in the mortar. This is necessary, because the graphite is more destroyed in the upper layer than below, and there may also possibly be imperfectly roasted portions here and there, which are thus brought into fresh contact with the graphite. After spreading out the mixture again in the capsule, which, if necessary, is painted with fresh reddle, it is again heated to redness, but more strongly than at first. Sometimes at the commencement, fumes, resulting generally from some remaining arsenic, may be smelled, but shortly not the least trace can be smelled, and after continuing the blast awhile, nearly all the graphite is destroyed, and the assay can be removed from the coal.

The only indication of a well-roasted assay is the absence of any odour of volatilizing constituents over the assay, which is glowing, and mixed with undestroyed graphite.

When the assay contains *sulphide of lead,* only a part of the sulphur combined with the lead is removed, and basic sulphate of lead remains. Moreover, when examining an ore dressed on the large scale for copper, if it contains *barytes* or *gypsum* and must be first roasted, it is impossible to remove the sulphuric acid combined with the lime or baryta, either by means of coal or graphite. When the ore contains an admixture of *calcite,* this is likewise altered during roasting into sulphate of lime, if metallic sulphides are present. If disregarded, these undecomposable sulphates may have a very injurious effect on the success of the assay during the reduction of the oxide of copper formed, since they are decomposed by fusion with alkaline fluxes, and give rise to the formation of sulphide of copper. During the fusion for black copper, therefore, regard must be had to this point, and the remaining sulphur be made harmless as there indicated.

The roasting is followed by

THE FUSION FOR BLACK COPPER.

The roasted substance, which may contain, besides oxide of copper, various metallic oxides and earthy matters, is charged for reduction with

100 to 150 milligr. soda,* according to the amount
of silicic acid (quartz) present,

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* The soda and borax-glass must be perfectly dry, or else so lively a motion occurs in the mass during the fusion that a mechanical loss can scarcely be avoided.

50 milligr. borax-glass, and
30 to 50 milligr. test lead.

If the substance contains sufficient lead, or if there is considerable antimony, bismuth, or tin present, as in *tetrahedrite*, *emplectite*, and *stannite*, and the roasting has been conducted with great care, no test lead is needed, because the reduction yields an easily-fusible alloy, from which the copper can be obtained pure. It is, to be sure, safer to add at least thirty milligr. test lead, even with plumbiferous substances, on account of the unavoidable formation of sulphate of lead.

The soda serves as a reducing agent for the oxide of copper and other easily-reducible oxides, and to slag off the quartz or silicates; the borax as a solvent for oxides of difficult reduction, as oxide of iron, manganese, and cobalt, and for many earthy ingredients; the test lead takes up the reduced copper, and forms an easily-fusible alloy, *black copper*, from which the pure copper can be separated in a short time.

The roasted substance and charge are well mixed in the agate mortar, transferred to the mixing capsule, and thence to a soda-paper cylinder, as in the silver assay. In this charge, however, there is less test lead, and it does not pour into the cylinder so readily, so that the ivory spoon-handle, or the spatula, must be used to push the charge in small portions into the cylinder, after which all adhering particles are brushed in. The closed cylinder is then placed in a coal crucible, or in an excavated coal, and a pure R. F., at first very feeble, and subsequently made stronger, directed upon it until all the paper is destroyed, and either various-sized metallic globules appear in the fluid glass bead, which is now to be regarded as a slag, or the reduced copper, united with the lead or other easily-fusible metals, appears as a button beside it. From this time the reducing flame is directed only upon the slag, the position of which is so changed by slowly turning the coal that the separate globules can unite to one metallic button. After several times altering the position of the slag with regard to this fluid button, so that every part which has been in contact with the coal appears free from metallic globules on coming into the opposite position, the blast can be stopped and the fusion regarded as complete. The metallic button is either taken out with the forceps the moment it solidifies, or is allowed to cool in the slag on the coal, and then separated from it between paper on the anvil, after which the oxidizable metals combined with the copper are removed with appropriate fluxes or by simple volatilization, according to the processes described hereafter under the metallic compounds. The button must be light bluish-gray or whitish, and have a weak metallic lustre.*

Slag which is gray or black when cold, and is quite free from metallic globules, which can be best ascertained with the magnifying-glass, can be thrown away, but if it contains metallic globules, or has a more or less red colour indicative of suboxide of copper, the assay must be treated some time longer in the R. F., and it is well to add about fifty milligr. of soda. Care must be taken to lose no slag when removing it from the black copper button, as it might be necessary to remelt it. As a rule, the assay is carefully examined with the glass after removing it from the coal, to see whether all the copper is reduced and melted to one button, so that the slag need not be previously separated from the black copper, in case a further fusion is required.

If the surface of the black copper button is very gray, or nearly black, and without any metallic lustre, it is nearly certain to contain more or less *sulphur*, resulting from *sulphate of baryta, lime*, or *lead* in the roasted substance, or from imperfect roasting. The first-named salts are decomposed by the soda during the reduction, forming at first only sulphide of sodium, which, however, afterward yields sulphur to the reduced metals and the copper. In case of imperfect roasting, the substance still contains sulphates of the metallic oxides, which are very easily reduced to sulphides. In case the admixture of earthy or metallic sulphates in the roasted substance is considerable, and the reduction is made *rapidly* at a high temperature, the black copper button is covered with a very brittle crust of sulphide of copper and lead; while if the reduction was made *slowly*, so as to afford the sulphur an opportunity to volatilize as sulphide of lead, the resulting button is sometimes tolerably free from sulphur. The metallic sulphides formed in reducing an assay containing a small quantity of earthy or metallic sulphates, are also destroyed by continued fusion in presence of sufficient lead, so that in most cases the black copper button is free from metallic sulphides.

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* All black copper free from antimony, bismuth, tin, and sulphur, is malleable; while that containing more or less of these bodies, or trifling quantities of arsenic combined with nickel and cobalt, is more or less brittle, and therefore the greatest care must be taken to avoid mechanical loss in removing the slag

In case the button is surrounded with a crust of sulphide of copper and lead, it must be very carefully separated from the slag between the paper, or, to avoid mechanical loss, the whole assay is melted on coal in the R. F., and the button taken out quickly as soon as it is solid. It is then melted on coal in the R. F., with about twice as much test lead and some borax-glass, and the borax bead beside the button treated with the R. F., until all the sulphur has volatilized with a part of the lead, leaving an alloy which shows a clean metallic surface on cooling. The button is then separated from the colourless glass, and refined with boracic acid according to *B*, *a*.

If the substance contained much *antimony*, and was therefore charged without test lead, then, in presence of sulphates, the sulphur is volatilized in combination with the antimony during the reduction, so that a copper button containing only antimony is obtained, which is refined according to *B*, *c*.

A copper button containing bismuth or tin is freed from a little sulphur by refining, and is described below in *B*, *b*, *a*.

b. *Ores, minerals, and products containing copper of oxide, or combined with chlorine; it being in the former case pure, or combined with acids or water, or slagged with earthy matters, or otherwise combined.*

Here are ranked *atacamite*, *cuprite*, *tenorite*, *melaconite*, *crednerite*, *cupreous manganese*; likewise all minerals containing sulphate, phosphate, carbonate, arsenate, chromate, vanadate and silicate of copper; among metallurgical products, all sorts of *slags* from copper smeltings; among artificial products, especially *colours* prepared from copper and *blue vitriol* with other *cupriferous vitriol*.

With the exception of the vitriols and the poor slags, a quantity of assay powder is prepared from the substance, and one ctr. weighed out and charged as follows, without roasting :—

100	milligr.	soda,
50	,,	borax-glass,
30–50	,,	test lead.

In case phosphoric acid is present, as in phosphate of copper, which is not entirely reduced by the above charge, twenty milligr. fine iron filings are added.

The addition of test lead is unnecessary for cupriferous slags containing not too little oxide of lead, as in certain refining slags; but if they at the same time contain protoxide of nickel, which is easily reduced and combined with the copper, it is better to add some lead. The substance is mixed with the charge in the agate mortar and fused just like the roasted substance (pp. 410–11). The soda acts partly as a reducing agent, and partly as a base for the non-reducible acids; the borax, as a solvent for the earthy matters and metallic oxides of difficult reduction, and it also prevents the soda from sinking into the coal in their absence; the test lead, as a protection against mechanical loss; the iron filings, to separate the phosphoric acid as phosphide of iron, which, indeed, combines with the black copper, but is separated from it simultaneously with the lead during the refining, described under *B, a.*

Blue vitriol, or other *cupriferous vitriols* [illegible] thus tre[illegible] sulphuric acid is red[illegible] sulphur, which, [illegible] again separated after reduction, and combines with the reduced copper to sulphide of copper, and this cannot be freed from the sulphur without loss. It is safer to dissolve two hundred milligr. of the vitriol over the lamp-flame in a porcelain vessel with water, convert the protoxide of iron into sesquioxide with a few drops of nitric acid at a boiling heat, precipitate the metallic oxides from the hot solution with potassa, and then wash the precipitate well on a filter, which is dried and ignited over the mortar. The oxides and the filter-ash are then mixed with one hundred milligr. soda, fifty milligr. borax-glass, and twenty to thirty milligr. lead, and treated as before for black copper.

In case of slags so poor that the copper in a centner can scarcely be separated and weighed as a pure button, fifty to eighty milligr. of gold, beaten thin, are added, and the copper then combines so perfectly with it that none can be detected in the slag after fusion. The fluid gold must, however, be carefully brought into contact with every part of the likewise fluid slag during the reduction. If the slag contains only protoxide of iron and suboxide of copper, the increase of weight in the button freed from slag gives the amount of the copper; but if oxide of lead was present, the button also contains lead, which must first be separated with boracic acid, as will be described in the separation of lead from copper. If besides suboxide of copper protoxide of nickel was present, this is likewise reduced, imparting to the gold a gray colour and making it harder and more brittle, while it would cause too high an amount of copper if disregarded. The button is therefore weighed and afterward treated on coal with borax, so as to keep the blue flame in contact with the borax, when the nickel is oxidized and dissolved, colouring the glass brown, while the copper ramains with gold, and its weight is again ascertained. The separation of the nickel is slow, but unattended by any loss of copper if carefully conducted.

B. Assay of alloys in which copper forms an ingredient.

a. Alloys of copper and lead.

Here belong the plumbiferous copper obtained by the reduction of the foregoing substances with the test lead, as well as the cupriferous lead obtained from the reduction of a roasted ore in the lead assay, *q. v.* Among metallurgical products are to be reckoned here the *cupriferous raw lead* obtained on the large scale, and the liquation discs, carcasses and dross from the liquation process.

.1 The compound of copper with lead obtained from the reduction of one ctr. of any cupriferous substance, if rich in copper, is thus separated:—

A shallow cavity, about four millim. deep and eight millim.

wide at the top, is bored in the cross section of a coal or in a coal capsule, in which an amount of vitrified boracic acid equal to the weight of the plumbiferous copper button is fused; then the button is laid beside it and fused as rapidly as possible, being covered with the blue flame. When this is done, the blue cone is made to cover only the boracic acid, but not the button, which should also be constantly in contact with the glass on one side and the coal on the other. A slight fault in the inclination of the coal may very easily cause the button to go under the glass, at once interrupting the oxidation, in which case the coal must be inclined toward another side and a strong blast used, so that the button may appear again. While the glass is covered with the blue flame, the lead is oxidized and at once dissolved in it. This process is continued until the button begins to assume a greenish colour, and at this moment a more spreading flame is caused to act on the glass, so that the remaining lead is slowly oxidized, while the copper is protected from oxidation, and the copper button does not then sputter. If the cavity in the coal is too small, or the blast stronger than is just requisite, the copper almost always sputters, even when still combined with a a little lead; the dimensions given above must therefore be observed, while the blast is just strong enough to keep the copper fluid and its surface bright. When the button shows perfectly the peculiar bluish-green colour of melted copper, indicative of the proper fineness, the process is stopped, the solidified button removed from the still soft slag with the forceps, and its qualities examined. It has a copper-red colour, can be hammered out without cracking, and when broken shows under the glass a true copper colour and a hackly texture, which can, however, only be determined in case of larger buttons, it is certain that the copper is free from foreign admixtures. If the slag on the coal is also transparent and only coloured yellowish with oxide of lead, there has been no chemical loss of copper, and the button may be weighed. Should the slag, however, show red streaks, or appear very red, this indicates a loss of copper, which can be very quickly determined as follows :—From boracic acid that is not saturated with oxide of lead, no lead can be reduced by a good R. F., while the dissolved oxide and suboxide of copper are very easily reduced to metal. It is only necessary, after removing the refined button, to treat this slag and the glass separated from the button for a time with the R. F., when it becomes transparent and yellowish on cooling, and the reduced copper appears in separate globules. These can be collected either by replacing the large button in the slag and fusing the whole with a powerful R. F. until all the small globules have united with the large one, which is then again removed from the slag; or the slag may be at once broken up between paper, and removed from the metallic particles by triturating and washing it in a porcelain dish. The little copper globules are then dried in the dish over the lamp, and weighed with the large button. If the glass contains also considerable oxide of lead, some lead is mingled with the reduced copper, which in this case is generally all in one button, and is then fused for a short time with the R. F. in a cavity on coal with a little boracic acid, when the lead is soon separated and leaves the copper pure. This is then weighed with the large button.

Should the assay substance contain more than an unimportant amount of silver, which has been ascertained beforehand by a silver assay, this must be deducted from the weight of the copper. In case the amount of silver in the copper button is to be determined, it must be fused with fifteen parts of test lead and cupelled according to p. 369, *et seq.*

2. In the lead assay, if the substance has been roasted before the fusion, cupriferous lead ores containing much copper yield plumbiferous copper, otherwise they yield cupriferous lead, if the lead prevails; likewise in assaying poor copper ores, etc., for copper, the addition of test lead causes only cupriferous lead. The refining of plumbiferous copper has just been described, and to ascertain the weight of the lead, it is only necessary to weigh the alloy obtained from the lead assay, and then to deduct the weight of the refined copper and silver from it. The refining of cupriferous lead from a lead or copper assay, or lead obtained in the large way, as *raw lead*, cannot be made in one period, on account of the length of the oxidizing process, but must be divided into:

α. a concentration of the copper,
β. the refining proper.

Both operations are performed with boracic acid as before, but if enough of this were employed at once to take up the oxide of lead formed, the remaining copper button would seldom be pure, since it would be concealed by the large quantity of slag before it was refined.

α. A concentration of the copper must therefore be first performed. The cupriferous lead from the lead assay is weighed and melted to a button in the R. F., with a little soda and borax-glass: this operation is unnecessary in case of the button from the copper assay. If lead, as raw lead, is to be examined for copper, one ctr. is weighed out and fused on coal, if not already in one piece. The cupriferous lead is then treated with its weight of boracic acid on coal, just as in the refining process above described, so long as it can be kept from passing beneath the glass and until little globules of reduced lead are observed. When the greater part of the lead has been thus removed, the process is stopped, and the button containing the concentrated copper freed from the glass on cooling. If the glass appears like a white enamel, as is almost always the case, there has been no loss of copper, and the

β. proper refining of the copper ensues, by which the concentrated plumbiferous copper button is refined according to p. 416. The lead is determined by deducting the amount of copper and silver, if any.

b. Alloys consisting of copper with iron, nickel, cobalt, zinc, and bismuth, either singly or with several of them at once, and containing frequently some lead, antimony, and arsenic.

In this class belong:

α. Bismuthiferous copper reduced in the assay of minerals containing bismuth and copper; *niccoliferous black copper* obtained by assaying certain copper-refining slags; the frequently very impure *black copper* and the *liquation residues* obtained in the treatment on a large scale of cupriferous lead matt.

β. The *black copper* obtained in the large way from copper ores free from lead.

γ. *German silver* and other compounds of copper and nickel, containing very little or no lead.

a. Refining of the copper containing bismuth, lead, or nickel, obtained from an assay, as well as the frequently very impure black copper obtained in the treatment in the large way of cupriferous lead matt.

The bismuthiferous copper obtained in the copper assay from minerals containing these metals, where no test lead was added, must be treated on coal alone in the O. F., until most of the bismuth has volatilized; then a little lead is added and the whole refined with boracic acid, when the trifling residue of bismuth likewise separates.

The black copper obtained in the large way is often very impure, and contains, besides copper and lead, iron, nickel, cobalt, zinc, antimony, arsenic, etc.; it is very brittle, and can be hammered only into easily-broken scales. One ctr. of it is melted on coal, with a little soda and borax-glass and twenty to thirty milligr. of test lead, to a button, which is then treated with one ctr. boracic acid, exactly according to p. 443 until either reduced globules of lead are observed in the glass, or the copper button is covered with a film of oxide, and can only be kept fluid with difficulty. During this process, lead, iron, antimony, zinc, arsenic, and other easily-oxidizable metals are oxidized, with some nickel; the metals which volatilize with difficulty combine with the boracic acid, while the others partly volatilize and partly combine with the acid; a part of the nickel, however, owing to its slight capacity for oxidation, remains obstinately with the copper and causes a thin film on its surface, hindering the refining of the copper. By continuing the oxidizing process, this film of protoxide of nickel is indeed taken up by the boracic acid and the rest of the nickel slagged off, but not without loss of copper. The remaining niccoliferous copper must therefore be fused with its weight of test lead and subjected to a new oxidation quite similar to the first. The surface is thus increased and the nickel oxidized with the lead almost without loss of copper. Should the slag, however, be red and some copper be reduced from it, after removing the refined button, this must be collected by triturating and washing the slag, and then dried and weighed. The nickel is not easily reduced unless the slag is saturated with oxide of lead and nickel, being usually only reduced afterward by using a very powerfully reducing flame. Such black copper being frequently rich in silver, the amount of this metal must be ascertained and deducted from the copper found.

After learning to refine such a black copper, it is easy to refine the plumbiferous and niccoliferous black copper button obtained by assaying a copper-refining slag. Should the lead present not suffice to separate all the nickel, a second addition is necessary.

β. Refining of the black copper obtained in the large way from copper ores free from lead.

This is generally combined with iron and occasionally with some zinc. *Brass*, being a similar alloy, although containing much more zinc, will be included here.

Any alloy of copper, zinc and iron can be assayed for refined copper like plumbiferous copper. A quantity of the metal is made fine and one ctr. weighed and melted on coal to a button with one or one-half ctr. of test lead, as the copper contains much or little zinc and iron; a little soda and borax-glass being added to the charge. This button is then treated with its weight of boracic acid just like any plumbiferous copper. The iron and part of the zinc oxidize with the lead, while part of the zinc is volatilized and the copper remains alone. Since the black copper in question sometimes contains a little nickel and cobalt, the remarks on the impure black copper above must be regarded here. Any copper in the slag may be obtained again as before described, and if there is an important amount of silver, it must be determined according to p. 417 and deducted.

γ. The determination of copper in presence of much nickel, as in German silver, packfong, etc.

The copper cannot here be determined accurately by refining with lead, since in slagging off so much nickel a considerable amount of copper is also slagged off. A method is given under the assay for nickel and cobalt by which both the copper and nickel in such alloys may be determined pretty accurately with the help of the blowpipe and the wet way.

c. Alloys of copper and antimony.

Here is included especially the *antimonial copper* obtained in assaying tetrahedrite for copper.

Antimony in combination with copper alone can be easily volatilized on coal in the O. F. without loss of copper. The copper button is placed in a cavity on natural coal, or a coal capsule, and kept in a melted state with the O. F., the point of which is occasionally directed a little to one side, so as to admit air freely to the assay. The antimony then volatilizes, leaving the copper behind. When there is much antimony, and the coal is burned away so that the metal lies too deep, it is sometimes necessary to stop the process and complete the volatilization of the antimony in a fresh cavity. The purity of the copper is indicated by its bluish-green colour while melted and its true copper-colour when cold, as well as by its malleability. Should these tests not be satisfactory, the oxidizing process must be repeated until it is quite pure, when it is weighed. The silver generally present, if it has not been already determined by a special assay, must be now determined, and if weighable, deducted from the weight of the argentiferous copper button.

d. Alloys of copper and tin.

Here are ranked the *alloy of copper and tin* obtained in assaying stannite for copper; and among artificial products, *bell, gun,* and *speculum metal,* as well as *bronze*.

Since boracic acid, when not combined with oxide of lead, melts with difficulty, and has no particular solvent action on binoxide of tin, it is advantageously replaced by a flux which is easily fusible and dissolves the binoxide. This flux is composed of—

100 parts by weight of soda,
50 „ „ „ borax-glass, and
30 „ „ „ silicic acid (quartz).

It is best to melt a quantity of this mixture in a platinum crucible and preserve the resulting glass in a well-closed bottle.

To refine stanniferous copper, about sixty milligr. of the above flux are melted to a bead on coal, and the alloy to be separated is laid beside it. For the compound obtained by reducing stannite the above amount is required, but for the other artificial products forty-five to fifty milligr. is enough. The bead and the metal are then fused with the R. F., so that the metal may assume a rotary motion, and the moment this occurs the flame is made somewhat more oxidizing and directed only upon the glass, but so as to cut off the access of air from it as much as possible. The button then begins to oxidize and the oxide of tin, which is mingled with protoxide of iron in the presence of iron, is immediately taken up by the glass. In case of copper from stannite, which through imperfect roasting is not quite free from sulphur, the sulphur is at the same time removed; but if there is too much sulphur, a loss of copper in the slag is unavoidable. It is then necessary to make a new assay. When, however, the metallic compound is such that the copper can be separated pure, the following points must be observed during the refining. During the oxidation of the tin the assay must be so held toward the flame that the alloy may always be in contact with the coal on one side and the glass on the other, so that no copper can be oxidized. The glass is capable of dissolving considerable oxide of tin, and the process is continued without interruption until it is completely saturated, which is indicated by the appearance of little specks of reduced tin here and there in the enamel-like glass. Upon this the process is stopped and the solidified metal removed from the still soft slag with the forceps, and transferred, with the trifling adherent slag, to another coal, where it is treated with sixty milligr. of the flux, just as before, until it begins to assume the colour of melted copper. When this appears, the glass is treated with a moderately strong and not too spreading R. F., until the metal beside it (to which, however, the air must always have free access) possesses the characteristics of pure melted copper. The blast is now immediately stopped, the button removed as before, and first examined as to colour and then as to malleability. If it has a true copper-colour, and does not crack when beaten out to three or four times its original diameter, it can be regarded is pure, and weighed; otherwise it must be treated with twenty to thirty milligr. of the flux as above, so as to refine it perfectly.

In this process of separation, which indeed requires some practice, care must be taken lest a part of the copper becomes oxidized and goes into the slag with the tin, which is, however, very easily perceived, because the suboxide of copper gives the glass a brownish-red colour when cold. Such a slag is treated a few moments with the R. F., to separate the copper, which is then united with the button by the side of the slag. The oxide of tin is not so easily reduced unless the glass is supersaturated with it. Some copper is always oxidized while removing the last portions of the tin, which by careful treatment amounts on the average to 0·3 milligr. for 25 milligr. of copper.

4. THE LEAD ASSAY.

The quantitative determination of lead with the blowpipe can be performed in two ways. The more complicated process is, however, only recommended for substances containing, besides the lead, copper, which is at the same time to be quantitatively determined. It consists in first removing as far as possible the volatile constituents, as sulphur, arsenic, and antimony, by roasting, and then fusing the roasted product with soda and borax-glass in a clay crucible, lined with charcoal paste, by which the oxides of lead and copper are reduced, while the earthy matters and non-reducible metallic oxides combine to a slag with the soda and borax, and the reduced lead and copper separate in larger and smaller buttons. Substances free from the volatile constituents above named are fused without roasting. The cupriferous lead, or plumbiferous copper, is then weighed and treated with boracic acid, according to pp. 416–417, when the copper remains behind. The simpler and shorter process consists in fusing all plumbiferous, and at the same time the cupriferous, substances unroasted, or only roasted in particular cases, in unlined clay crucibles with metallic iron and a fluxing and reducing agent,

whereby the lead separates in a button, and the earthy matters, with the non-reducible oxides and the sulphides, are slagged off.

The plumbiferous *minerals, ores, and products* can be classified into—

A. Those containing the lead as sulphide.

B. Those containing the lead as chloride and as oxide, free, or in the form of slag, or combined with acids.

C. Those containing the lead in the metallic state, combined either with selenium, or with other metals.

A. Assay of Minerals, Ores, and Metallurgical Products containing the Lead as sulphide.

FIRST METHOD.

The substances for which this method is especially suitable are:—all the minerals containing sulphide of lead which are at the same time cupriferous, as *bournonite, cuproplumbite*, etc.; and among metallurgical products, particularly *cupriferous lead matt* and *plumbiferous copper matt*.

One centner of the assay powder is freed from volatile constituents by careful roasting on a clay capsule painted with reddle, as in the copper assay, p. 407, with addition of charcoal-dust. Besides the coal, about fifty milligr. of sesquioxide of iron are added to substances which *sinter*, or fuse together easily, as bournonite, etc.

When all of the coal has been consumed at a low red heat (sulphide of lead may easily be volatilized by a stronger heat), and no more fumes of volatile constituents can be observed, the capsule is removed and the ore rubbed in the mortar and mixed with twice its volume of fresh coal-dust, after which it is replaced in the clay capsule and roasted the second time. When the fresh coal takes fire, the presence of volatilizing constituents is quickly tested by smelling, and in case a considerable quantity of them is detected, the coal is allowed to consume at a moderate red heat, after which it is again rubbed in the mortar, and if necessary roasted a third time with coal. Although seldom occurring, the third roasting is unavoidable when the substance to be assayed for lead contains metallic sulphides and arsenides, which are decomposed with difficulty. If after the second addition of coal the substance evolves no odour while the coal is glowing, or only a feeble odour of sulphurous acid, the assay may be regarded as well roasted. After the coal has been slowly burnt out from the ore, lustrous particles of undecomposed sulphides must not be present, but the whole mass should have a dull, earthy appearance, and must lie in a porous state on the capsule.

The roasted assay is charged, for the reduction of the oxide of lead in it, with—

100 milligr. soda (anhydrous),
30–40 „ borax-glass.

The soda, in common with the coal which surrounds the charge during the fusion, acts as a reducing agent on the oxide of lead, chiefly through the formation of carbonic oxide, and at the same time takes up the sulphuric acid remaining with the roasted oxides, especially the oxide of lead, forming sulphide of sodium. Any other reducible metallic oxides are separated in the metallic state, and the others are brought to the lowest stage of oxidation and then go into the slag. The borax prevents the soda from sinking into the coal lining, and dissolves the non-reducible oxides. The amount given is generally sufficient to form a perfectly fluid slag; but in case many earthy matters, or bodies to be converted into slag, are present, it is well to increase the amount of borax-glass to fifty milligr.

Both of the fluxes are mixed with the roasted ore in the mortar, and then poured into the mixing capsule and transferred to a soda-paper cylinder, as in the preceding assays. The cylinder is not closed by pressing the corners of the empty part upward after rolling it together, as in the copper or silver assay, but downward, so as to lie upon the filled portion, in order that the wrapped-up assay may rather have a hemispherical shape.

It is then placed in a clay crucible lined with coal, which must, however, be thoroughly dried as soon as it is made. The assay is then covered with so much fine charcoal-dust that, when the clay-roasting capsule which serves as a cover is laid in an inverted position upon the crucible, the whole space between the capsule and crucible is filled. During this operation the crucible is set on a tripod of iron wire.

The coal-holder is provided with an artificial or natural coal, but only an opening for the flame is bored in this with the borer without cutting it out, as is necessary for the roasting, and the slit in the coal-holder is closed with the screen of sheet-iron, *h*, Fig. 48. The platinum wire, on which the crucible is to rest, is set in without the shield, since the crucible protects the coal from being too rapidly burned through. The full crucible is now placed in the ring, Fig. 74, so as to leave a space between it and the coal all around, and especial care must be taken that the lower part of the crucible may be distinctly seen through the opening at *a*, and not only the point of it. The clay capsule is placed upon the crucible, and the whole covered with a natural or artificial coal prism, which fits into the coal-holder, being held by the projecting sides, and has on its inner surface a cavity, *o*, and an opening, *p*, four millim. wide.

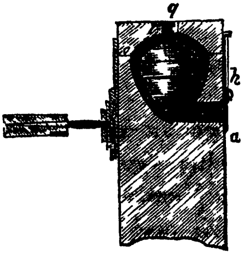

Fig 74.

After arranging everything thus, and providing the blowpipe with a tip having not too narrow an aperture, a strong O. F. is directed through the hole *a* in a horizontal direction, so that the blue point of the flame is still seen outside. The temperature increases so rapidly from below that after a few minutes a little flame of burning carbonic oxide is seen at *q*, Fig. 74. If the coal is not too hard or dense, in which case only can the proper heat be produced, the most refractory assay will certainly be fused after blowing uninterruptedly for eight minutes. After that time has passed, the blast is stopped, the coal cover removed, and the wire seized with the pliers at *n*, so as to raise the covered crucible and set it on the tripod, or other stand, to cool.

In a successful assay there should be no coat of oxide of lead visible on the upper side of the coal cover near the opening, which would indicate so strong a heat that some lead was volatilized. Moreover, the fused assay must lie as a perfect ball at the bottom of the lined crucible, and be easily removed with the forceps from the almost uninjured coal lining. It happens very rarely that the lead forms one button, but it generally lies in the slag in several separate buttons, large and small. The slag is broken coarsely between paper on the anvil, and poured into a porcelain dish, when the larger buttons, free from slag, are picked out and the slag washed from the remaining lead buttons by carefully triturating it with the agate pestle and pouring it off with the water, repeating the operation until all of the slag is removed. The remaining lead is then dried in the porcelain vessel. Should the previously removed larger buttons not be free from slag, they must be beaten out on the anvil, cleaned with water, dried with the others, and weighed.

When the roasting was carefully performed, the reduced lead can only contain *copper*, *silver*, *bismuth*, and *antimony*. The *copper* is found by fusing the lead buttons together on coal with borax-glass and treating them with boracic acid, according to p. 415. The *silver* is then determined by cupelling the resulting copper button with fifteen times its weight of test lead. Should *bismuth* be present in the substance assayed, as well as lead and copper, it is best to make two assays; one serving to determine the copper in the alloy, as well as the combined weight of the bismuth and lead, while the other is performed to determine the amount of the lead and the bismuth in the following way:—The larger buttons produced are flattened out and fused, together with the smaller ones, in a porcelain vessel, with about twenty times their weight of bisulphate of potassa, over the spirit-lamp, until the alloy is oxidized and combined with the sulphuric acid. More of the salt is added if necessary. The fused mass is then further treated as will be described under the quantitative bismuth assay, and the sulphate of lead collected on a filter, dried thoroughly, weighed, and the lead calculated; one hundred parts of the sulphate contain 68·3 parts of lead. By deducting this and the weight of the refined copper obtained from the other assay, the amount of bismuth is obtained, at least approximately.

When the assay contains *antimony*, only the greater part of it is removed by roasting, and a little remains and is reduced. The antimony in such an alloy can be qualitatively detected by treating it on coal in the O. F., when it forms a white coat of oxide of antimony. The quantitative determination could only be made in the wet way, and would not be very feasible on account of the small quantity present. Antimony is oxidized by the fusion with bisulphate of potassa, but not separated from the lead.

Finally, it must be remarked that in treating by this process substances containing sulphide of lead, the amount of lead obtained is always from one to three per cent. too low, because during the roasting the lead is chiefly converted into basic sulphate, which is, indeed, decomposed by the soda, but in consequence of the formation of sulphide of sodium some sulphide of lead also results and goes into the slag. The loss can even increase if the fusion is continued too long at a high temperature, because then some lead is liable to be volatilized.

SECOND METHOD.

The substances which can be assayed by this method are:—compounds containing sulphide of lead; among ores dressed on the large scale, *galenas* and all lead ores mixed with other metallic sulphides and arsenides; among metallurgical products, especially *lead matt*, and *plumbiferous cadmia* (*Ofenbruch*), as well as *lead slags* and *Rohschlacken* (*raw slags*).

One centner of the assay powder is poured at once into a clay crucible, provided it contains little or no antimony. If it contains a notable amount of antimony, it is previously roasted in a clay capsule, either alone, or, if necessary, with addition of fifty milligr. sesquioxide of iron, but no charcoal-dust, at a gentle heat, according to p. 407, until the fumes of oxide of antimony cease. Then the requisite fluxing and reducing agents are added as follows:—

1. Metallic iron in the form of wire, the thickness of a moderately coarse knitting-needle; according to the amount of metallic sulphides present, and the proportion of sulphur

they contain, the piece employed varies from twenty-five to fifty milligr., and it is laid directly on the weighed substance in the crucible.

2. An alkaline fluxing and reducing agent (Plattner's flux), consisting of equal equivalents of anhydrous carbonates of soda and potassa, with borax-glass and starch-powder in the following proportions :

10 parts by weight of carbonate of soda,
13 „ „ „ „ „ „ potassa,
5 „ „ „ „ powdered borax-glass,
5 „ „ „ „ dry starch-powder.

These ingredients are carefully mixed in a spacious mortar and then preserved in a well-closed bottle. If no mortar is at hand, the ingredients, when fine enough, can be mixed by shaking them together in a corked bottle. Three hundred milligr. of this easily-fusible flux are poured directly upon the substance and the iron in the crucible, and the whole is covered with three heaped spoonfuls of decrepitated common salt, or about six hundred milligr.

Fifty to eighty milligr. of pure silver in one button are added to substances containing only a little lead, in order to collect the latter.

During the fusion of the assay, the iron serves to separate the sulphur and arsenic, the latter being, however, generally volatile; the alkaline carbonates, with the borax, serve to form the slag and dissolve the earthy matters, with such metallic sulphides as are not decomposed by iron, as well as to take up the greater part of the sulphide of iron formed; the starch acts as a reducing agent, in common with the carbonates of the alkalies, while the salt, being very fluid when melted, and having no tendency to combine with the slag, serves as a cover, so that the separate reduced lead buttons can more easily unite into one. The substance to be assayed might be mixed with the flux, but then little globules of lead are liable to come to the surface of the slag, and the union into one button is retarded. When, however, the substance consists chiefly of substances that must be slagged off, such a mixture is to be recommended.

For the fusion a coal is prepared in the coal-holder, and the crucible is then set on the wire ring without any cover, the perforated charcoal cover is placed above it, and a strong O. F. directed into the opening *a* in a horizontal direction, so that the point of the blue flame remains just outside, while, for the most part, only the glowing gaseous products of combustion enter the hollow coal. The crucible must not be touched directly by the flame, or else the high temperature at that spot will cause it to be attacked and perforated by the alkalies. The heat spreads quite rapidly when the coal is not too dense, and after blowing five or six minutes at the most, the assay is perfectly fused. Even after the first minute the noise caused by the lively evolution of gas from the action of the substances in the crucible can be heard, and so long as this is distinct the blast must on no account be strong, or the evolution of gas will become too lively and cause the charge to overflow. After the noise ceases, however, a strong blast must be kept up for at least one or two minutes, and the point of the flame directed very particularly toward the lowest point of the crucible, if a correct quantitative determination of the lead is desired. After stopping the blast, the coal cover is removed and the coal-holder struck on the side with the handle of the forceps, so that the little lead globules which may still be scattered about the border of the crucible, or the surface of the slag, may sink and combine with the main button. The slag should be as fluid as water at this time. The crucible is then lifted out and set on its stand to cool for some minutes, until it can be held in the fingers, when it is carefully broken with the hammer on the anvil, and the lead, with the iron adhering to it, separated from the slag. The lead is then held with the forceps on the anvil so that the iron is on top, and this, which is sometimes surrounded by sulphide of iron in case of very pyritiferous ores, is detached by a few light strokes of the hammer. Any adhering portions of slag are removed from the lead by hammering it gently between moistened filter-paper, after which the button is weighed.

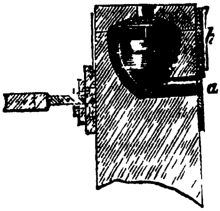

Fig 75.

In this second process the formation of alkaline sulphides cannot be avoided, and therefore, in spite of the addition of iron, a little sulphide of lead also goes into the slag, and the result is always semewhat too low, at in the first process, and likewise in the dry assay on the large scale.'

If the substance is supposed to be argentiferous, the lead button should be cupelled and the weight of the silver deducted. If the substance assayed cnosisted of pure galena, the silver thus found weighs the same as that obtained by a special silver assay, but it is too little if the substance contains argentiferous pyrites, or other argentiferous sulphides.

A trifling amount of sulphide of copper in the plumbiferous substance goes into the alkaline slag, but if considerable of it is present, or the copper is in the oxidized state, and there is, moreover, a lack of metallic sulphides, more or less copper always goes into the lead. In this case it is necessary to treat the lead with boracic acid, according to p. 417, and to deduct the weight of the resulting copper from that of the cupriferous lead.*

When the substance contains only one to ten per cent. of lead, it is difficult to separate the lead button from the iron, so as to weigh it accurately. In this case fifty to eighty milligr. of silver are added, in small clippings, or in one or several buttons, and the lead combines with this to a button, which is so large that it can be easily separated from the iron. The increase of weight gives the amount of lead.

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* In cases where the presence of considerable antimony necessitates preliminary roasting, and a trifling quantity of copper may be present, this can be prevented from going into the lead by mixing the roasted assay first with one hundred milligr. of sulphur in the crucible, and heating it to low redness in the square coal, until the blue flame ceases. The oxide of copper formed during the roasting is thus transformed into sulphide, which goes into the slag.

B. Minerals, Ores, and Products containing the Lead in the state of chloride as well as of oxide, free, as slag, or combined with acids.

Here are included *chloride of lead*, as well as combinations of oxide of lead with *phosphoric, arsenic, sulphuric, carbonic, acetic, vanadic, molybdic, tungstic*, and *chromic acids*; further, *litharge, abstrich*, and *cupel bottoms*, and all sorts of *plumbiferous glasses*.

These compounds may, indeed, be assayed according to the first method on p. 424, the roasting being only performed when other metallic sulphates are present, or when metallic sulphides and arsenides are intermingled; the second method is, however, far simpler, and equally reliable.

One centner of the substance is poured into a clay crucible, and twenty-five to thirty milligr. of iron wire added in one piece. Three hundred milligr. of the fluxing and reducing agent, p. 429, and twenty-five to thirty milligr. of additional starch-powder, are then mixed directly in the crucible with the substance, with the ivory spoon-handle or the small iron spatula. The mixture is then made as compact as possible by striking the crucible carefully upon the table, and three heaped spoonfuls, or about six hundred milligr., of common salt added as a cover.

An addition of fifty to eighty milligr. of pure silver is required for slags and substances containing little lead. The fusion is performed in precisely the same manner as before described. The alkaline flux serves to decompose the lead salts, and, in common with the starch, to reduce the oxide of lead and other reducible ingredients, while it forms, in common with the borax, the necessary slag. The iron serves chiefly to prevent any sulphide of lead from going into the slag, in case it should be formed from sulphate of lead present.

If the substances belonging here are not free from a trifling amount of copper, the assay can be sulphurized before charging it with the flux and reducing agent, according to the note on p. 431.

C. Minerals containing the Lead in the Metallic state, combined either with Selenium or with other metals.

In this class belong *clausthalite, tilkerodite, zorgite, lehrbachite, altaite, nagyagite*, and *mullerite*.

These minerals can be most simply assayed in an unlined clay crucible. One centner of the finely-pulverized mineral is mixed directly in the crucible with three hundred milligr. of the fluxing and reducing agent, twenty-five to thirty milligr. of iron wire added in one piece, as a precautionary measure, the whole compacted by striking the crucible carefully against the table, and then three heaped spoonfuls of salt added as a cover. The fusion is made just as before. The compounds of lead with selenium or tellurium are decomposed, alkaline selenides or tellurides being formed, and the lead liberated. If the mineral likewise contains metallic sulphides, then sulphides of sodium and potassium are also formed. After the fused assay is cold, the crucible is broken, and the separated lead detached from the iron and slag. The lead need only be cupelled in order to determine any silver or gold combined with it. When considerable copper is present, the lead button is unavoidably cupriferous and must then be refined to determine the copper.

5. THE BISMUTH ASSAY.

Bismuth generally occurs in nature only in the native state, but it is also found in combination with tellurium and sulphur, both alone and in combination with sulphides of copper, lead, and other metals, and some rare minerals; likewise in the oxidized state, partly free and partly with carbonic, phosphoric, and silicic acids.

In metallurgical products it sometimes forms a constituent of cobalt speiss from the manufacture of smalt, and of nickel speiss from the smelting of bismuthiferous nickel ores, etc. In purifying with dilute hydrochloric acid, the impure oxide of tin ore roasted on the large scale, and containing bismuth, which is converted into oxide by the roasting, basic chloride of bismuth is obtained, which is rendered more or less impure by other substances, as earthy matters, fine particles of oxide of tin, etc. The same salt is obtained in extracting bismuth by the wet way from the silver-refining hearth.

The *minerals, ores,* and *products* to be assayed for bismuth can, therefore, be classified thus:

A. Such as contain bismuth in the metallic state; either mixed only with earthy substances, or arsenides of cobalt, nickel, and iron; or else chemically combined with tellurium or silver.

B. Those in which the bismuth occurs as sulphide, either alone or chemically combined with other metallic sulphides or arsenides.

C. Those containing the bismuth as oxide, free, or combined with carbonic, phosphoric, or silicic acids, etc., and possibly mingled with oxides of copper, nickel, and cobalt, or their salts; or else containing the bismuth combined with chlorine.

A. Assay of Minerals, Ores, and Products containing the Bismuth in the metallic state, either mixed only with earthy substances, or arsenides of Cobalt, Nickel, and Iron; or else chemically combined with Tellurium or Selenium.

In this class belong all *cobalt* and *nickel ores*, dressed on the large scale, and containing an admixture of native bismuth; among minerals, *native bismuth* disseminated in earthy gangues, *tetradymite*, *joseïte*, and *wehrlite;* among metallurgical products, the *cobalt* and *nickel speisses.*

One centner of the powdered substance is weighed out, and, if necessary, prepared for the fusion as follows:—Cobalt and nickel ores dressed on the large scale and containing these metals combined with so much arsenic, that they give a sublimate of metallic arsenic in the closed tube, and therefore contain more arsenic than the combination $(Ni, Co)^2 As$, must be freed from this excess. When the substance to be assayed consists of such an ore, the weighed quantity is poured into a clay crucible which is placed on a proper support, and is to be subsequently used for the fusion also. The crucible is then put into a square coal fixed in the coal-holder, and in which a ring of iron wire is substituted for the similarly-shaped platinum wire, just as described under the fusion of a lead assay, p. 425. The crucible is then covered with a clay capsule and the whole with a perforated coal cover, the blowpipe flame being directed through the hole in the coal-holder as before described, so as to bring the interior of the coal and the crucible to a dull redness. The excess of arsenic then escapes, and the substance, when rich in metallic arsenides, sinters together more or less strongly.* When the fumes cease to ascend, the blast is stopped, the wire, together with the crucible, removed with the pliers, as described in the lead assay, p. 425, and allowed to cool with the cover on, so as to prevent access of air and roasting. After removing the excess of arsenic by this gentle ignition, the assay is ready for the fusion.

When the substance is cold, a piece of iron wire weighing about thirty milligr. is added, which is indispensable for the thorough decomposition of the sulphide of bismuth and the saturation of the metallic suphides and arsenides, which cannot be decomposed by the fusion. Further, in order to collect the bismuth separated during the fusion, and to separate it after the fusion from the simultaneously liberated metallic arsenides, and the excess of unaltered iron, so that there may be no mechanical loss of the brittle bismuth, fifty to two hundred milligr. of pure silver, according to the quantity of bismuth expected, is added in fine clippings or a button, which have been exactly weighed out. During the fusion the bismuth forms an alloy with the silver, and if there is three or four times as much of the latter as of the former, this alloy on cooling is much less brittle than pure bismuth: after deducting the weight of the silver, it shows the amount of bismuth, provided no other metals have been reduced at the same time.

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* Should copious fumes of arsenic be evolved during the preliminary test in the closed tube, it is well to perform the ignition outside of the laboratory, under a chimney with a good draught or elsewhere. Should there be no opportunity for this, the excess of arsenic can also be removed in a tube sealed at one end, as will be described in the cobalt and nickel assay for substances under *B*, which must be "arsenicized."

Lead might be used in place of silver, since one part of bismuth with about four of lead gives a likewise malleable alloy, but the results would be less exact, because with a large proportion of bismuth in the substance so much lead must be added, that several per cent. of it might easily be lost as sulphide in the slag, or by volatilization, and the amount of bismuth found would be proportionately smaller.

To slag off earthy matters, as well as any metallic oxides of difficult reduction, and such metallic sulphides as are reduced neither by iron nor by alkalies in a clay crucible, and also to decompose the compounds of bismuth with sulphur and tellurium, the fluxing and reducing agent, p. 429, may be used. Three ctrs. of it are added to the substance with the iron and silver already in the crucible, and, if the substance is in powder, the whole is mixed with the spoon-handle, after which the crucible is carefully struck against the table and the level surface covered with three heaped spoonfuls of salt. When the substance has sintered or fused together in the preparatory operation, the above mixing cannot be performed, and is unnecessary, because in this case there are few or no ingredients to be slagged off.

The crucible then has been filled with—

100 milligr. of the substance to be assayed,
30 ,, ,, iron, or thereabouts,
50–200 milligr. silver,
300 ,, flux, and
the requisite covering of salt.

It is now placed on the platinum or iron wire ring in the square coal, just as described for the lead assay, p. 425, and the whole covered with a square perforated coal. The fusion is then conducted, observing the directions given for the lead assay fusion, so that it may be completed in five or six minutes at the most. When finished, the coal cover is removed, and the crucible with the perfectly fluid assay lifted out with the forceps and placed on its stand to cool. As soon as it can be held with the fingers, it is carefully broken on the anvil, and the metallic button at the bottom freed as much as possible from adherent slag. The button will be either perfectly round and free from adherent iron, when the substance contained a notable amount of metallic arsenide; or it will not be quite spherical, owing to adhering iron, which is possibly covered with some sulphide of iron, when the substance was free from arsenides. In the former case, careful examination will show that the button consists of two separate alloys, one white and consisting of bismuthiferous silver, and one gray, and composed of arsenides of cobalt, nickel, and iron. It is placed between paper on the anvil, or, if none of the arsenides are to be lost, in the steel mortar, and the arsenides separated from the silver by a few careful blows of the hammer, which is generally very thoroughly effected. The alloy may even be flattened somewhat when the bismuth is combined with three to four times as much pure silver. It is, however, impossible to remove the trifling quantity of slag and the traces of arsenides from its surface, and the button must therefore be fused, in a cavity on coal with a little borax-glass, for a few moments, in a feeble R. F., but only until the surface becomes bright, when it is immediately allowed to cool, and can now be separated from the slag, which only adheres to one side, by a few blows on the anvil. This must of course be carefully done, and no bismuth volatilized by too long a blast. The slag last detached from the button is added to the metallic arsenides, in case they are to be further examined for cobalt and nickel, as will be described in the quantitative assay for those metals.

After weighing the bismuthiferous silver, and deducting the weight of the silver, the bismuth is ascertained, unless the substance itself contained a weighable amount of silver. Should this be suspected, the bismuthiferous silver button is cupelled with one ctr. of test lead, weighed, and the corresponding cupellation loss reckoned from p. 376; this will show whether there was a weighable amount of silver, which must be deducted. The amount of silver can also be determined by a direct assay for silver.

Should some metallic iron be found on the side of the metallic button, this is likewise separated between paper, or in the steel mortar, and the bismuthiferous silver button fused as before on coal with borax-glass, to detach perfectly the adhering slag. The weight of the bismuth and any silver originally present in the substance is then determined as above.

B. Minerals in which the Bismuth occurs as sulphide, alone, or chemically combined with metallic arsenides, or with other sulphides.

This division includes *bismuthinite*, *karelinite*, *emplectite*, *wittichenite*, *schapbachite*, *aikinite*, *chiviatite*, *kobellite*, and *grünauite*.

With the exception of *kobellite*, which contains sulphide of antimony and must be roasted, according to p. 407, the composition of these minerals admits of their fusion, without any further preparation, in a clay crucible with suitable alkaline fluxing and reducing agents; a bit of metallic iron being added to prevent the possible slagging of sulphide of bismuth; also the amount of silver necessary to collect the separated bismuth.

After the roasting, if necessary, and again after sulphurizing the roasted product, according to p. 439, when copper is also present, one centner of the mineral is charged in a clay crucible with—

30 milligr. of iron wire, or thereabouts;
50–200 „ pure silver, in clippings or buttons;
300 „ of the flux and reducing agent used for lead assays, etc.

When not sintered together during the sulphurizing process which may have followed the roasting, the whole is mixed with the aid of the spoon-handle, compacted somewhat by striking the crucible against the table, and covered with three heaped spoonfuls of salt. The fusion is then conducted just as with the substances of the first class.

On breaking the crucible, the round button of silver and bismuth is found at the bottom, and beside it the iron added, which is generally coated with sulphide of iron. The metallic button separated from the iron on the anvil is fused a few moments with borax-glass on coal, until it shows a bright surface, and when cool is freed from the trifling adherent slag, and weighed.

Some of the above-named minerals contain other metals besides bismuth, viz., copper, silver, lead, and antimony. When there is little copper, and this is present as sulphide, little or none goes into the metal; the antimony can, for the most part, be separated by the preliminary roasting; when, therefore, a special assay has shown only a very trifling amount of silver, the increase of weight consists of bismuth and lead, with possibly copper, provided the substance contained lead.

To determine the amount of each of these metals, the metallic button is fused in the platinum capsule, over the spirit-lamp, with twelve to fifteen times its weight of bisulphate of potassa, and small portions of this salt are added from time to time, if required, until the metallic button has disappeared. The capsule is then placed in a porcelain vessel, and heated with distilled water until the fused salt is dissolved and the sulphates of lead and bismuth have separated in a pulverulent form (p. 255). After settling some time, the whole is filtered, or the clear solution of sulphates of potassa and silver is decanted from the residue into another vessel with the aid of a glass rod, fresh water is added, stirred, and the whole allowed to settle, after which the water is again decanted.* The residual sulphates of lead and bismuth are covered with water containing some sulphuric acid and heated to boiling, when the sulphate of bismuth dissolves easily, especially after adding a few drops of nitric acid, while the remaining sulphate of lead can be collected on a filter and washed. It is then dried thoroughly, weighed, and the amount of metallic lead in it calculated; 100 parts of sulphate of lead contain 68·3 parts of lead. The bismuth is obtained by the difference, in case the button was free from copper. Should copper be present, as shown by the more or less blue colour of the mass obtained by the fusion with bisulphate of potassa, the bismuth must be precipitated from its solution by warming it with carbonate of ammonia, filtered, and when dry reduced with the addition of silver in a clay crucible, by means of the flux already mentioned. This method is, however, only to be recommended when the solution shows a distinct blue colour.

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* The silver can be extracted from the decanted solution, after diluting it by adding salt solution, and proceeding as described in p. 400.

Although the determination of lead and bismuth by the preceding method is not quite exact, and in presence of copper is very complicated, it at least gives approximately true results. The amount of lead obtained is, moreover, incorrect when a little antimony is present, because this metal, although oxidized by fusion with bisulphate of potassa, is not separated from the lead.

If a notable amount of silver has been found by a special assay, this must be deducted together with the weight of the silver added.

Example.—By a special assay for silver in a substance containing silver, lead, and bismuth, 15 °/₀ of silver has been found, including the cupellation loss; that is, 15 milligr. in 100 milligr. of the substance. In the assay for bismuth 100 milligr. of silver were added, and an alloy weighing 175 milligr. obtained. Deducting 115 milligr. for the silver, there remain 60 milligr. of bismuth and lead. From the separation with bisulphate of petassa 48 milligr. sulphate of lead, or 32·8 milligr. lead, are obtained; then 60—32·8=27·2 milligr. gives the amount of bismuth, and we have :—Ag.=15 °/₀, Pb =32·8 °/₀, Bi=27·2 °/₀.

C. Minerals, Ores, and Products containing the Bismuth as oxide; free, or combined with Carbonic, Phosphoric, or Silicic Acids, etc., and possibly mingled with Oxides of Copper, Nickel, and Cobalt, or their Salts; or else containing the Bismuth combined with Chlorine.

Here are included, among minerals, chiefly the following: *bismuth ochre, bismutite, eulytite,* and *hypochlorite;* among artificial products, the *basic chloride of bismuth* obtained by purifying, with dilute hydrochloric acid, the bismuthiferous oxide of tin ore roasted on the large scale, and also by the extraction of bismuth in the wet way from the mass of the silver-refining hearth, etc.

If the minerals named are free from disseminated metallic oxides, which are easily reducible, they may be at once charged and fused with the flux and reducing agent; but if they contain copper, nickel, or cobalt, in the oxidized state, either free, or combined with acids, they must first be converted into sulphides by ignition in a crucible with sulphur, according to the note on p. 431, or into arsenides, as will be described in the cobalt and nickel assay, in which latter case the amount of nickel, cobalt, and copper can be at the same time ascertained.

One centner of the properly prepared substance is charged in a small clay crucible, just as for the substances of the first and second classes, with—

25–30	milligr.	iron wire,
80–100	„	pure silver,
300	„	flux and reducing agent (p. 429).

The whole, being mixed in the crucible with the spoon-handle, and compacted by striking it on the table, is covered with three heaped spoonfuls of salt, and fused as the preceding substances of the first and second class. The result of the fusion is a bismuthiferous silver button, either with adherent iron, which is sometimes surrounded with sulphide of iron or speiss, or together with metallic arsenides (speisses), which have taken up the iron. The metallic button, carefully separated from the iron or speiss on the anvil, is fused with borax-glass on coal, to give it a bright surface, when it is freed from the adhering slag and weighed. By deducting the silver added, the weight of bismuth is obtained.

In examining the basic chloride of bismuth obtained from impure tin ores, which has been washed away as a basic salt and collected in settling-pits, some tin is also obtained in the alloy, when the chloride was not free from disseminated particles of oxide of tin. This is immediately perceived when melting the bismuthiferous button with the borax, as its surface is covered with a thin film of oxide, instead of becoming bright. In this case it is necessary to dissolve the button in nitric acid and separate the residue of binoxide of tin, the amount of which must be ascertained. For this purpose the solution of silver and bismuth in nitric acid is diluted with water and warmed, so that the previously very finely divided oxide of tin may adhere together somewhat and be separated by filtration. After washing and drying this, it is cautiously ignited in the platinum capsule and weighed; 100 parts of the binoxide of tin contain 78·6 parts of tin. By deducting the weight of the tin from the impure bismuth, the amount of bismuth is ascertained. (The proportion of tin thus obtained is, however, smaller than would result from a direct assay for tin, because some of the tin is slagged off, especially in presence of metallic sulphides.)

6. THE TIN ASSAY.

Cassiterite is the ore from which tin is made on a large scale, and on which the tin assay is principally founded. However simple the determination of the percentage of tin in the same may be, in the absence of foreign substances, still the assay becomes quite complicated when other reducible compounds are present, which is often the case, for they must be separated from the ore before the fusion, by treating it in the wet way.

In the quantitative tin assay, therefore, the minerals, ores, and products in which this metal forms an essential ingredient, can be classed as follows:—

A. Those which contain the tin either as sulphide, or as oxide mixed with sulphides and arsenides;

B. Those which contain the tin as oxide, free from sulphides and arsenides;

C. Those which contain the tin as metal, alloyed with other metals.

A. Assay of Minerals, Ores, and Products that contain the Tin either as sulphide, or as oxide mixed with sulphides and arsenides.

This class includes not only the tin pyrites or stannite, which is the only mineral in which tin occurs chemically combined with sulphur and other sulphides, but also the dressed tin slimes, which contain the tin as oxide, it is true, but in which very frequently small quantities of sulphides and arsenides are present, notwithstanding the roasting both before and after the dressing. Of the products belonging in this class, Mosaic gold must be mentioned, which consists of tin combined with its maximum of sulphur.

When a substance in this class is to be assayed for tin, one centner of the prepared assay powder is weighed out, and then the volatile ingredients driven off by roasting. A description of the method of roasting would be superfluous here, as the roasting of a substance to be assayed for tin is performed exactly in the same way as that of a copper ore, with coal-dust (p. 407, *et seq.*).

The roasting is quickly finished if the substance to be roasted contains among the volatile ingredients only sulphur, or sulphur with a trace of arsenic, or only a small percentage of sulphur and arsenic, which latter is sometimes the case with the tin slimes that have been roasted and dressed on a large scale. If, however, it contains much arsenic, the roasting with charcoal is longer, and is to be continued until there is not the slightest trace of the odour of arsenic noticeable, when charcoal is added, and the substance brought to a red heat. The dressed tin slimes, if they have already been roasted for dressing, require only one roasting, but it is necessary to roast the other substances, which contain the tin combined with sulphur, or as oxide mixed with sulphides and arsenides, two or three times with charcoal powder.

By this process, if it is well done, the sulphur and arsenic are driven off from the substance containing the tin, which is, perhaps, mixed with pyrite, mispickel, chalcopyrite, stibnite, bismuthinite (bismuth glance) or bismuth, blende, wolframite, etc., and, of the other metals, tin, if not already present as oxide, and the copper, iron, bismuth, and zinc are oxidized. Those metals, besides arsenic, which are capable of forming acids, and which are volatilized during the roasting only with difficulty, or not at all, viz., a part of the antimony, molybdenum, tungsten, and titanium, remain behind as acids.

The proper appearance of a well-roasted tin ore is very similar to that of a properly-roasted lead ore. The roasted ore must give no odour while still mixed with charcoal powder and heated to redness, and must not, when rubbed in a mortar after complete combustion of the coal, show any shining particles of sulphides or arsenides. It must also be in a very porous state when in the roasting capsule.

If a well-roasted tin ore, consisting, for example, of the oxides of tin, iron, manganese, bismuth, and copper, were immediately reduced, there would only result, even with the best reducing fluxes, a brittle and gray button of tin that would weigh too much. This would happen, because not only the oxides of copper and bismuth with that of tin are reduced, but a part of the oxide of iron is also reduced to metal and alloyed with the tin, owing to the action both of the large amount of charcoal present and of the reduced tin upon that oxide. As these disadvantages cannot be remedied by the dry way, it is necessary to dissolve out, by means of hydrochloric acid, the oxides of iron, manganese, bismuth, and copper that are mixed with the roasted tin ore.* The method is as follows :—

The well-roasted ore is put into a small porcelain dish, and hydrochloric acid poured upon it until the acid stands about 0·5 c.m. above the powder. The dish is then placed upon the igniting ring, which should be about sixty millim. above the flame, and the wick pushed down into the lamp, so that only a small flame remains, which heats the dish quite gently. The dish should be covered with a watch-glass.

This digestion is continued uninterruptedly for four or five minutes, and care taken that the acid does not boil too strongly. In the course of at most five minutes, in which time all the oxides of iron, manganese, bismuth, copper, and zinc, as well as any acids of antimony that might be present, are dissolved, the ring with the dish is turned away from the flame, and the whole allowed to cool.

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* BERTHIER recommends to boil the raw powdered tin ore a few minutes, with a sufficient quantity of aqua regia, to dilute the solution with water, to collect the residue upon a filter, and wash it well, by which the ore is freed from both the iron and from the other injurious ingredients. It is then only necessary to dry the filter and take out the contents, to burn the paper and ignite the whole in a porcelain dish or clay crucible, in order to burn off any sulphur that might have been separated during the treatment of the ore with aqua regia. Since, however, not a small quantity of sulphur is separated by this treatment from a tin ore that contains a large admixture of pyrites, and as this sulphur is apt to leave behind, after the ignition, small quantities of metallic sulphides which would injure the reduction of the oxide of tin, it is better to roast this kind of ore, and then treat it with hydrochloric acid.

The watch-glass is then removed from the dish, and freed by porous paper from any adhering acid. The residue consists either of oxide of tin or cassiterite alone, or maybe mixed with tungstic and titanic acids, for an admixture of wolframite and titanic iron is decomposed in such a way that the above-mentioned acids are separated. The insoluble powder may be freed in two ways from the clear, yellow, or green-coloured solution: first, by diluting with water the solution of the oxides, filtering, washing the residue, and burning the filter. This method must be employed in the assay of stannite or tin pyrites, and also for tin ores that are free from bismuth, so that no separation of a basic salt is to be feared when water is added; second, and in most cases, the solution, after the residue has settled, can be removed with the aid of a small glass pipette, and three or four times as much pure water added in its place. The water, however, must be dropped very carefully upon the sides of the dish, so that the powdered ore on the bottom will not be disturbed, nor the lighter particles of the same mixed with the water, because, otherwise, time will be required again to separate them. If the dish is now warmed, any portion of the solution that has remained behind will unite with the water, and the dish being inclined to one side, can be separated from the heavier powder with the aid of a small pipette, perfectly clear, if it is not rendered slightly cloudy by basic chloride of bismuth. If it happens that very small particles of the earthy portion of the powder float upon the surface of the water, and settle only with difficulty, the opening of the pipette must be kept as much as possible below the surface, so that the particles may not be lost by being drawn into the pipette. In order, then, to free the powder from the adhering water, the dish is put upon the ignition-ring, and heated until the powder is perfectly dry.

The entire operation, including the solution, requires but a quarter of an hour, when filtering is not necessary, and with proper care no loss of tin is to be feared. It is easy to understand that the protoxides of iron and manganese, which are chemically combined with the cassiterite, and which even in a dark-coloured tin ore scarcely amount to two per cent., are not removed by this operation. Since, however, these small quantities of the oxides are slagged off, and only a slight trace of the protoxide of iron is reduced to metal and alloyed with the tin, this inaccuracy, that can scarcely be noticed upon the balance, is not regarded.

If tin slimes are to be assayed for tin, which have already been roasted in the reverberatory furnace to effect an almost complete dressing, and which only contain a small percentage of sulphur and arsenic, the roasting may be omitted and they may be treated directly with aqua regia, as described by Berthier (*vide* note, p. 444), in the same way as the roasted ore, with hydrochloric acid.

After the substance to be assayed for tin has been freed from the constituents or ingredients that hinder the production of a pure tin, either by roasting and treating with hydrochloric acid or by aqua regia alone, the reduction of the oxide of tin is effected. This may be done, with the employment of proper fluxes, in two ways: either in a lined clay crucible, as in the reduction of the oxide of lead in roasted lead ores (p. 425), or in the same kind of crucible not lined, as for a lead assay according to the second method (p. 429).

a. The reduction of the prepared tin ore in a clay crucible lined with charcoal.

The charge for this fusion consists of—

100 milligr. perfectly dry soda, and
30 ,, borax-glass.

This charge is weighed out, while the ore, after having been treated with hydrochloric acid and washed with water, is drying, and is mixed with the same in the agate mortar. The mixture is then packed in a cylinder of soda-paper, in the same way as a roasted lead ore, and fused in a covered clay crucible lined with coal, as is described in detail on p. 425. The fusion of a tin assay requires from eight to ten minutes. After cooling, a button is found upon the bottom of the crucible, which consists principally of slag, and in this is inclosed the reduced tin, seldom in a single globule, but oftener separated into several large and small buttons. These buttons are, as in the lead assay, cleaned by rubbing and triturating the slag with water, and are then dried. As the slag from a tin assay is generally so constituted, in the absence of many earthy ingredients, that it dissolves quite easily in boiling water, the tin may be separated from it in the following way: The button consisting of slag and globules of tin is broken up, either between paper on the anvil or in the steel mortar, as much as the globules of tin permit, and the mass is transferred to a small porcelain capsule, water added, and the whole placed over the lamp. When the slag is dissolved, the fluid is carefully poured off, and the moist residue, which contains finely-divided particles of charcoal that have separated from the slag, is stirred up with the pestle, water again added, so that all the lighter portions of the slag that remain may be separated from the globules of tin. If, in consequence of the ore containing quartz, the slag is insoluble in water, it is only necessary to add some acetic acid, warm the whole, and then clean the globules of tin by washing with water. They may then be dried in the capsule over the lamp. The purity of the resulting tin is proved partly by the magnet, and partly by its colour and malleability.

If the roasting and separation of the oxides of iron and copper, as well as of the acids of antimony, which may be combined with metallic oxides, have been carefully done, the tin from the reduction, if the proper amount of heat has been employed, will be pure and of the right weight. If, however, the roasting and digestion of the ore in hydrochloric acid has not been continued long enough, the tin, after reduction, will be brittle when copper and antimony are present, and if it contains even a small amount of iron, will be malleable it is true, but when finely divided and brought under water, it will be attracted by the magnet, and its weight will certainly be too high. If the ore to be assayed for tin contains quantities of tungstic or titanic acids, these acids, as above mentioned, cannot be separated from the ore by means of hydrochloric acid. During the reduction of the oxide of tin, however, they mostly go into the slag, so that they are to be regarded as harmless. If, on the contrary, the amount of tungstic acid present is considerable, a part of the same is easily reduced, and the tin that results in this case is rendered impure by tungsten, which cannot be prevented.

Cassiterite, or tin-stone, occurs in *stockwerks* deposits, and veins in and with granite, gneiss, mica-schist, clay, talc, calcite, and many other gangues; it is found also accompanied by iron and copper pyrites, mispickel, native bismuth or bismuth glance, stibnite, blende, wolframite, molybdenite, ochre, magnetite, etc., and it is often so finely disseminated through some of these gangues, that no certain result can be obtained from a blowpipe assay, because the percentage may be too insignificant. In this case, after the ore has been finely pulverized and weighed, the earthy admixtures are to be removed by careful washing, and at least two quantitative tin assays made from the slimes thus obtained, in which the heavier tin ore is concentrated, after it has become perfectly dry and its weight has been ascertained. After weighing the tin obtained, it must be examined as to its purity, and the percentage calculated for the crude ore.

For example:—Suppose there was obtained from 5 grm. = 50 ctr. or 5,000 milligr. of the finely-pulverized ore by careful washing (performed in a beaker glass of the proper size with the aid of a glass rod), a quantity of slime, that weighed when dry 700 milligr.; from these 700 milligr. which were well mixed and rubbed together in an agate mortar, two tin assays were made. If there resulted from each of these assays 1·5 per cent. of tin, the 700 milligr. of slime would contain $\frac{700 \times 1\cdot5}{100} = 10\cdot5$ milligr. of tin. If the washing was carefully done, these 10·5 milligr. constitute almost the entire amount of tin in the 5 grm. = 5,000 milligr. of crude ore mentioned above; therefore, in 100 of this ore there are

milligr., or per cent., of tin.

b. The reduction of the prepared tin ore in a clay crucible not lined with charcoal.

The fusion of the properly purified tin ore can be made in a shorter time if a clay crucible is employed that is not lined, the ore being charged with an easily-fusible flux of the proper sort. For the tin assay in general is to be recommended, according to Winkler, the addition of some agent for collecting the reduced tin. In the blowpipe assay silver may be used, which is added to the tin ore in a finely-divided state,* in an exactly-weighed quantity of fifty to eighty milligr., according to the purity of the ore.

There are also added three hundred milligr. of Plattner's flux, as well as fifty milligr. of starch. The whole is then mixed with the handle of an ivory spoon, the mass being rendered more solid by carefully tapping the crucible upon the table, and covered with three heaped spoonfuls—about six hundred milligr.—of salt. The assay charged in this way is then fused in a square coal, as in the lead assay, p. 430.

After the fusion has lasted for five, or at the most six minutes, there is obtained: first, a perfectly fluid slag, which becomes light-gray when cold; and second, a metallic button, consisting of silver and tin, which is malleable, and the weight of which, after deducting the amount of silver, gives the amount of tin in the substance. Upon dissolving the button in nitric acid, the tin remains as oxide, and the silver may be obtained again in a pure state.

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* This finely-divided silver can be easily prepared by decomposing chloride of silver with metallic zinc. The dry chloride is fused in a porcelain crucible, water added when cool, a few drops of hydrochloric acid poured into it, and a piece of clean zinc laid upon the chloride of silver. In a short time the chloride is changed throughout to a gray mass of metallic silver, which must be pressed and rubbed, while moist, first with water containing hydrochloric acid, and then well washed with pure water and thoroughly dried. It can then be preserved for future use.

B. Assay of Minerals and Artificial Products which contain the Tin as Oxide.

The minerals and artificial products that belong in this class must be assayed for tin according to different methods, and are divided, therefore, as follows:

a. Those which are free from the oxides of iron and admixtures of metallic sulphides and arsenides.

b. Those which contain beside oxides of tin, silicates of the protoxide of iron and earths.

a. Assay of minerals and artificial products which contain the tin as oxide, free from the oxides of iron and admixtures of metallic sulphides and arsenides.

In this class belong, among minerals, the pure oxide of tin; and among the metallurgical products, zinnasche and enamel.

As a matter of course, with these substances the roasting before the reduction of the oxide of tin is omitted, and also the treatment with hydrochloric acid, provided no oxide of iron or copper, or acids of antimony, are present as admixtures. It is only necessary to weigh out one hundred milligr. of the perfectly dry, finely-pulverized cassiterite, or of those products which do not contain silica, and to charge the same with—

100 milligr. soda, and
30 „ borax-glass.

The reduction is then performed in a clay crucible lined with charcoal, as for the substances belonging under the former class.

In the determination, however, of the amount of tin in enamel, which is a combination of silica and oxide of tin, it is necessary to charge one hundred milligr. of the same with—

150 milligr. soda, and
30 „ borax-glass;

so that the silica may combine with the soda, while the oxide of tin is reduced. As enamel, however, often contains oxide of lead, which is also easily reduced, the tin obtained in this case is not pure, but is alloyed with lead. Such a combination cannot, it is true, be separated in the dry way, but this can be done with nitric acid, in which the lead is dissolved, while the tin remains behind as insoluble oxide. It is then only necessary to wash this oxide well with water on a small filter, dry, and ignite strongly in a platinum spoon. The amount of metal can be easily calculated from the weight of the ignited oxide; 100 equivalents of oxide of tin contain 78·6 of metallic tin.

The reduction of the substances that belong to this subdivision can also be made in a clay crucible not lined with coal, in the same manner as for the substances in the division *A*, p. 450.

b. Assay of products which contain the tin as oxide combined with silicates of the protoxide of iron and of the earths.

To this class belong principally the tin slags, or the slags from the smelting of tin ore, which are distinguished as follows:—

a. Slags (*Steinschlacken*), which are obtained from the direct smelting of the tin ore (cassiterite); they often contain besides the oxides of tin and unaltered particles of cassiterite, a considerable quantity of globules of tin, for which reason they are repassed through the furnace.

β. Slags, which result from the second smelting of the above-mentioned slags; they are, it is true, either free from globules of tin or at least much freer from them than the above, but they still contain more or less of chemically combined oxide of tin, as well as some unaltered particles of cassiterite mechanically mixed.

These slags, the former as well as the latter, cannot be assayed according to the methods described above, on account of the large percentage of protoxide of iron present, which is combined with silicic acid and sometimes partly with tungstic acid, because there would result a button of tin containing iron, and consequently a false percentage. As hydrochloric acid cannot be used to separate the protoxide of iron, because the combination of the silicates is not always perfectly decomposed by this acid, there being danger also of bringing tin into the solution, another made of treatment must be employed.

If the slags contain globules of tin, several small pieces must be pulverized in an iron mortar, the powder well mixed together, and two to five grammes weighed out, according as the globules of tin are small or large. The larger and free globules of tin are then removed with the forceps, and the powder rubbed fine in an agate mortar, in small portions at a time, the pure slag being each time separated, by elutriation with water in a porcelain dish or beaker glass, from the heavier particles of metallic tin. This alternate elutriation with water and pulverization of the residue of slag is carried on until all the slag is separated. The particles of metallic tin are then dried in a porcelain dish, weighed along with the other globules of tin that were first separated, and the percentage calculated. The elutriated slag is collected on a filter, dried, and prepared by a fusion with bisulphate of potash for the reduction of the oxide of tin that it contains. If tin slags are to be assayed, which are free from an admixture of globules of tin, only a small quantity is rubbed fine in an agate mortar, elutriated also with water, because it can only be used for the assay when in an extremely fine condition, dried, and prepared like the preceding, by a fusion with bisulphate of potash, for the reduction.

For this purpose one ctr. of elutriated and dried slag is carefully weighed out. Then fifteen to eighteen times its quantity (1·5—1·8 grammes) of bisulphate of potassa is fused at a low heat in a platinum capsule over the spirit-lamp, and the weighed slag, as soon as the fluid salt is perfectly quiet, gradually added in small quantities, the heat being increased at the same time. This fusion is continued until no more undecomposed particles of slag are to be seen in the glowing and fluid mass, and no bubbles of gas arise.* By this means the silicates are decomposed, the bases are for the most part dissolved (the protoxide of iron as sesquioxide), and the silicic acid remains with the oxide of tin. Tungstic acid is also left behind, if present in the slag. The fused salt is then dissolved in water with the aid of heat, the platinum capsule along with its contents being put into a porcelain dish containing water, to which a few drops of hydrochloric acid have been added, so that all the sesquioxide may go into solution. The residue is collected on a filter, washed with hot water, dried, and the filter burned. This residue contains all the tin as oxide, and is now ready for the reduction. For this operation it is mixed with—

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* It is of advantage to cover the platinum capsule with a piece of closely-fitting thin foil, in order to prevent any mechanical loss and an unnecessary evolution of sulphuric acid.

150 milligr. soda, and
30 „ borax-glass,

the mixture put into a cylinder of soda-paper and fused in a clay crucible lined with charcoal, as described for a substance in the division *A*. A malleable button of tin almost free from iron is then obtained, if the fusion with bisulphate of potassa has been properly conducted, and if the slag was sufficiently fine. If the slag contained a noticeable amount of tungstic acid, which could not be separated by means of bisulphate of potash, the tin will contain a little tungsten, because the tungstic acid is also reduced, and the tungsten easily combined with the tin. The amount of tungsten, however, is seldom important.

The reduction of the residue obtained from the fusion with bisulphate of potash can also be made in a clay crucible not lined with brasque, with the addition of silver, according to p. 449.

C. Assay of Alloys that contain Tin as an ingredient.

In this class are included bell and gun metal, bronze, as well as every combination of tin with lead, bismuth, zinc, and antimony. Since, however, the quantitative determination of the tin in the last-named alloys, and also in bronze when it contains lead, as is often the case, is very inaccurate in the dry way before the blowpipe, but generally easy and more exact in the wet way, the tin assay proper is limited to bell and gun metal, and bronze free from lead.

The separation of tin from copper has been described in detail under the copper assay (p. 421), where, however, the copper only is regarded, and no attention paid to the tin. If it is purposed to determine the amount of the tin at the same time, care must be taken to lose none of the glass, which contains all the tin, because the tin must be reduced from it.

When the percentage of tin only is to be determined with the blowpipe in an alloy consisting of tin and copper, the tin must first be oxidized, according to the method described on p. 422, and in this state carefully separated from the copper by means of a glass made up of soda, borax, and silica. The glass containing the tin is then pulverized, mixed with about fifty milligr. of soda, put in a cylinder of soda-paper, and fused in a lined clay crucible, as in the ordinary tin assay. After the fusion, the tin generally contains a trace of copper, because, during the separation of the two metals, a very small portion of the oxide of copper is also generally taken up by the glass. The resulting tin is then weighed, and the percentage calculated.

7. THE COBALT AND NICKEL ASSAY.

Among the minerals containing chiefly cobalt there are only a few free from nickel, while, on the other hand, the minerals and ores rich in nickel, as well as many furnace products, such as the various speisses, which occur as intermediate products, partly in the manufacture of smalt, and partly in smelting niccoliferous and cobaltiferous silver ores, contain more or less cobalt.

There will, therefore, be few cases where either of these metals is to be quantitatively determined alone in a substance, but they must generally be both determined at once. Since the method to be followed is the same for the assays of both metals, their assays will be described in common, as well as the methods by which any copper, lead, or bismuth present can be simultaneously determined.

Cobalt and nickel being difficult to fuse, cannot be separated from their compounds with the blowpipe in a pure metallic state, like silver, gold, lead, etc.; but both can be determined with comparative ease and great accuracy, by separating them in combination with arsenic. The method rests upon the fact, stated in the qualitative examination for iron in metallurgical products (p. 214 *et seq.*), that by an oxidizing fusion, with borax on coal, of compounds of metallic arsenides containing cobalt and nickel, the arsenides more easily oxidized than arsenide of nickel can be successively slagged off, according to their oxidizability; so that from a substance consisting of arsenides of nickel, cobalt, iron, and after separating the arsenide of iron and any excess of arsenic, a compound of the arsenides of nickel and cobalt remains, in which each of these metals is combined with a definite quantity of arsenic. By then slagging off the arsenide of cobalt, the quantity of arsenide of nickel can be determined, and, consequently, the proportions of both these metals can be calculated.

If one centner of such a substance, containing but little iron, is fused with some borax-glass in a shallow cavity on coal, with a powerful R. F., or within the blue flame, until the compound assumes a rotary motion, and then the O. F. is directed immediately upon the fused bead of borax, the arsenide of iron oxidizes first, and, when a suitable and not too high temperature is employed, covers the free surface of the metallic compound with a thin crust of basic arsenate of iron; this is carried into the borax-glass through the motion of the metal, and is immediately dissolved, while part of the arsenic combined with the iron and other metals volatilizes, as can be perceived from the odour of the fumes. If the blast is stopped when the surface of the metallic compound becomes bright, and is no longer covered with a crust of oxide, while arsenic fumes still arise, and the solidified button is removed from the still soft slag, the latter will have a black colour, and a little of it fused with borax on platinum wire in the O. F., reacts only for iron; the button, however, has become coated with an almost black crust of oxide while cooling. If much arsenide of iron is present, one fusion with borax will not suffice to separate it, and it is then necessary to treat the button with fresh borax, until it becomes bright and begins to give off arsenic fumes: after each fusion the solidified button is removed from the saturated borax-glass and thrown into water, so that the still adherent slag may crack off and be easily separated by rubbing between the fingers or in paper. When this button, freed from slag, is kept in fusion in a shallow cavity on coal, with a feeble R. F., so as to remain fluid and with a bright surface, until the fumes of the arsenic cease to rise, a constant compound results, consisting of arsenides of nickels ($Ni^4 As$ = 39·27 As and 60·73 Ni), and arsenide of cobalt ($Co^4 As$ = 38·46 As and 61·54 Co). By further fusing the button with borax, just as in separating the arsenide of iron, the arsenide of cobalt is oxidized to basic arsenate of cobalt, and dissolved in the borax, while only a slight arsenic odour can be perceived if the excess of arsenic had been fully removed. Upon employing the same heat as was used in oxidizing the iron, the surface of the button appears bright, but if the temperature sinks somewhat, a film appears occasionally, which passes quickly away; the blast being interrupted, the borax-glass shows a smalt-blue colour, when pinched out and drawn into threads. The separation of the cobalt is much slower than that of the iron, and until it is all gone the solidified button is always covered with a black crust of oxide, as after the separation of the iron. The brightening of the button continues, however, on adding fresh borax and using a proper heat, so long as the arsenide of cobalt is present; but when all the cobalt is separated, and the arsenide of nickel begins to oxidize, a film of basic arsenate of nickel forms, which moves slowly about on the surface, and is finally dissolved by the borax; and this lasts as long as the oxidizing process is continued, and the borax-glass can take up the arsenate. To observe this exactly, however, it is necessary to know the proper temperature; when the heat is too strong, stronger than in the oxidation of the cobalt, even pure arsenide of nickel may easily show a bright surface when fluid. If the blast is stopped so soon as the phenomenon above described can be distinctly perceived, and a part of the glass immediately pinched out and slowly raised, remaining still connected with the main portion, it appears generally rather violet than blue against the daylight, provided it is not too strongly coloured with cobalt. If all the cobalt had been separated before, the glass would only appear pale brown, but this brown with the blue of the cobalt causes the violet colour. On the surface of the remaining arsenide of nickel, beside the purple glass, is seen an apple-green film of basic arsenate of nickel, which also indicates that only Ni^4 As remains. The Co^4 As is completely slagged off, while the Ni^4 As retains none of the arsenic from it, and therefore both metals can be quantitatively determined in their compounds. The loss of nickel is only unimportant, with proper care. Even in cases where much nickel is present, the whole surface of the button must be covered with the apple-green film, and the borax appear slightly coloured with oxide of nickel, in order that the loss shall amount to 0·5 milligr.

The nickel is not so easily determined in compounds containing copper, which is generally combined with sulphur and remains with the arsenide of nickel, while the other ingredients usually occurring with arsenide of cobalt and nickel can be separated before determining those metals. Such a compound is the speiss which separates as a special product in smelting argentiferous lead ores, when silver ores containing nickel, cobalt, and copper are smelted with them. It consists (p. 199) chiefly of arsenides of iron, nickel, and cobalt in varying proportions, mingled or combined with more or less of the sulphides of copper, lead, iron, etc. To assay such a substance for cobalt, nickel, and copper, the copper must likewise be combined with arsenic, and any lead or bismuth separated by means of iron. The arsenides of iron, cobalt, and nickel can then be successively separated from the copper by an oxidizing fusion with borax, and under certain conditions with considerable precision.

The arsenide of copper is transformed into Cu^6 As=28·31 As and 71·69 Cu, while the arsenides of cobalt and nickel pass into the tetrarsenides. While separating the more easily oxidizable arsenides of cobalt and nickel with borax, the proportion of the arsenic to the copper remains constant only so long as the arsenide of copper is combined with at least an equal amount of arsenide of nickel. As soon as more of the arsenide of nickel is slagged off, some arsenic volatilizes from the copper, and it is impossible to determine the amount of pure copper from the weight of the remaining arsenide of copper, or to calculate the amount of the arsenide of nickel slagged off. A very satisfactory result is, however, obtained if, after separating the arsenide of cobalt and determining the cobalt, there is added to the compound of $Ni^4 As$ and $Cu^6 As$, *in which the amount of copper must not exceed that of the nickel*, a gold button sixty to one hundred milligr. in weight, according to the amount of copper. The slagging of the arsenide of nickel is then continued, until all the arsenide of nickel and the arsenic combined with the copper are completely removed, and the alloy of gold and copper shows a bright surface, with the bluish-green colour peculiar to melting gold, as well as copper, while the button remains bright on cooling.

As arsenide of nickel is slagged off very slowly with borax, salt of phosphorus is best employed, and the process performed just as in the separation of the arsenide of cobalt from arsenide of nickel. The outer flame is directed immediately upon the glass, with only a feeble blast, and the air is allowed to have access to the button; the arsenide of nickel is gradually converted into basic arsenate and dissolves in the glass, which it colours pure yellow. When the first portion of salt of phosphorus is saturated, the button is taken out, cooled in water, freed from slag, and treated with a fresh portion, until its surface ceases to be covered with a film of oxide and begins to show the bluish-green colour; the last portions of arsenide of nickel are then preferably separated by means of borax, in order to prevent any slagging of the copper, while at the same time the still remaining arsenic is volatilized. If the cupriferous gold button shows a clean, metallic, lustrous surface, and can be beaten out when cold without cracking, it is weighed, and the copper determined from the increase in weight.

To determine the nickel, the amount of arsenic belonging to the copper, which was combined as $Cu^6 As$ with the $Ni^4 As$, must be calculated, and also the amount of $Ni^4 As$ remaining after deducting the arsenide of copper. From this the amount of nickel can be easily calculated, as shown in the example given below.

For the sake of brevity in calculating the amounts of the metals from the weighed arsenides, their composition is taken as follows:—

Co^4 As .... 61·5 % cobalt,
Ni^4 As .... 60·7 % nickel,
Cu^6 As .... 71·7 % copper.

A proof that nickel and cobalt, treated as described, form constant compounds with arsenic, in which 4 atoms Co or Ni are combined with 1 atom As, is obtained by treating the purest *skutterudite* for the cobalt and pure *niccolite* (*copper nickel*) for the nickel.

Skutterudite ($Co^2 As^3$) contains 78·95 As and 21·05 Co, with perhaps 2 % Co replaced by Ni and Fe. One ctr. of this mineral, powdered and mixed with about 50 milligr. soda and 15 milligr. borax-glass, is treated in a soda-paper cylinder on coal with the R. F., until all the metallic parts have united to a button, when the trifling amount of iron goes into the flux and most of the arsenic volatilizes. Any sulphur present goes into the flux, causing it mostly to sink into the coal. The button is removed from the coal, freed from slag, and fused on another coal, in a not too shallow cavity, with the R. F., until no more arsenic volatilizes. There remains a compound weighing about 33 milligr. Too strong a flame may cause mechanical loss through sputtering; the flame should be just sufficient to keep the button fluid and bright.

The remaining compound of arsenic and cobalt gives the clearest proof that, of the 78·95 milligr. of arsenic present, 2½ parts = 65·8 milligr., have been volatilized, and only ½ part = 13·16 milligr., remains with the cobalt and the little nickel, in a combination of

1 atom = 38·46 As, with
4 atoms = 61·54 Co.

Disregarding the nickel, the remaining button would contain:

100 : 61·54 : : 33 : 20·3 milligr. cobalt;

a result nearly corresponding with the composition of pure skutterudite as given above. By treating this button with borax on coal in a feeble O. F., adding fresh portions of borax until it was a little coloured with nickel, and then weighing the remaining button, the amount of $Co^4 As$ would be found by the loss in weight. Suppose that 2·5 milligr. $Ni^4 As$ remain after this treatment, then 33 − 2·5 = 30·5 milligr. $Co^4 As$ have been separated, containing $\frac{61·5 \times 30·5}{100} = 18·7$ milligr. (or p. c.) Co.

To calculate the nickel in the remaining 2·5 milligr. $Ni^4 As$, we have $\frac{60·7 \times 2·5}{100} = 1·5$ milligr. nickel.

Pure *niccolite* consists of

1 atom = 56·4 As,
2 atoms = 43·6 Ni.

Generally, there is about 1 p. c. of foreign bodies, as Co, Fe, Pb, Bi, and S. On treating 1 ctr. with 50 milligr. borax-glass, first with the R. F. and then with the O. F., until the arsenide of nickel begins to oxidize, then stopping the blast immediately, freeing the button from slag, and fusing it alone on coal with a moderate R. F., until the arsenic fumes cease, a button weighing only about 71 milligr. results, and it has therefore lost about 29 %. The chief loss is caused by the nickel yielding half of its arsenic. Since niccolite contains 56·4 % As, about 28 parts volatilize and the rest remains with the nickel as $Ni^4 As$, a constant compound, consisting of—

1 atom = 39·27 As,
4 atoms = 60·73 Ni.

Remembering that the niccolite generally contains a few foreign ingredients, and therefore lost by the previous treatment 29 p. c. of its weight, we find this loss to consist of—

27·4 = As combined with the nickel,
0·6 = As combined with cobalt and iron,
1·0 = Co, Fe, Pb, Bi, and S.
———
29·0

The remaining 71 % of arsenide of nickel must then correspond nearly to the composition $Ni^4 As$. Pure niccolite contains 43·6 p. c. Ni, and therefore, diregarding the trifling impurities, there is in the remaining 71 p. c. arsenide of nickel 71 − 43·6 = 27·4 p. c. As, and the following ratios result:

$$71 : 27{\cdot}4 :: 100 : 38{\cdot}6 \text{ As},$$
$$71 : 43{\cdot}6 :: 100 : 61{\cdot}4 \text{ Ni}.$$

These correspond nearly with the composition of pure $Ni^4 As$, making allowance for the impurities and the trifling loss of nickel in the borax.

CLASSIFICATION OF THE COBALTIFEROUS AND NICCOLIFEROUS SUBSTANCES ACCORDING TO THEIR CHEMICAL CHARACTERS.

The substances to be assayed for cobalt, nickel, and in many cases also copper, lead, or bismuth, may be classified, with respect to the methods to be pursued, as follows:—

A. Those containing cobalt and nickel in the metallic state combined with arsenic, frequently mixed with other arsenides, and sometimes with trifling quantities of metallic sulphides, but free from copper.

B. Those containing cobalt and nickel, with perhaps other metals, partly combined with arsenic and partly with sulphur, or else with sulphur alone, but containing an admixture of more or less metallic sulphides, or earthy constituents.

C. Those in which cobalt and nickel in the oxidized state, combined with arsenic or other acids, or with other metallic oxides, and sometimes also water, form a prevailing, or only an accessory ingredient.

D. Such as consist chiefly of oxides of cobalt and nickel alone.

E. Those consisting of alloys, or metallic sulphides and arsenides in which more copper than nickel is present.

A. Assay for Cobalt, Nickel, and, if necessary, Bismuth, in Minerals, Ores, and Products, which contain the Cobalt and Nickel in the metallic state combined with Arsenic, and are frequently mixed with other metallic arsenides, and also sometimes with very trifling quantities of metallic sulphides, but are free from Copper.

Here belong, among minerals, compounds of cobalt and nickel combined with arsenic; among metallurgical products, the *cobalt speiss* from the smalt-works, and the *nickel speiss* obtained directly by smelting cobaltiferous nickel ores or niccoliferous cobalt ores.

With substances containing no bismuth, or where it is not necessary to determine the trifling amount of it present, the first operation of fusing the assay with a suitable flux can be performed in two ways: either in a coal crucible, as in the silver assay, or in an unlined clay crucible, as in the lead and bismuth assays. By the latter method the mechanical loss attendant on mixing, wrapping up, and fusing the assay is entirely avoided, and with cobaltiferous substances containing little or no iron there cannot well be any oxidation of the cobalt during the fusion, while the presence of earthy constituents also does no harm; on the other hand, it cannot be used when arsenide of cobalt prevails in the assay, on account of the difficult fusibility of this compound.

The fusion on coal or in a coal crucible is performed on one ctr. of the substance, which is mixed in the agate mortar with—

50 milligr. soda, and
15 „ borax-glass,

transferred to a soda-paper cylinder and then treated with a pure, moderate R. F., until the flux has become a slag and the metallic particles have united to a button.

The soda not only forms an easily-fusible slag with the borax, furthering the rapid union of the metallic parts and the separation of any sulphur, while it takes up some iron as protoxide, but it also prevents the easy oxidation of the cobalt when a pure R. F. is used. The resulting metallic button is freed from slag by cooling it in water, and then all iron must be removed from it before the Co^4 As and Ni^4 As can be formed. The button is therefore fused in a cavity on coal with a moderate R. F., when, if no iron is present, it speedily shows a bright surface and assumes a rotary motion. In this case it is kept fused until the fumes of arsenic cease, when the blast is stopped and the button weighed; the weight gives the sum of the $(Co\ Ni)^4$ As. During the operation, by which any trifling admixture of bismuth is also volatilized, the blast must be just strong enough to keep the button fluid and bright, since it may easily spit with too high a heat. If the button contains iron, it does not show a bright surface, but is quickly covered with a film of oxide, when borax-glass must be added and the usually trifling amount of iron slagged off, according to p. 455, until the button shows a perfectly bright surface. A little cobalt is unavoidably lost by slagging with the last portions of the iron; but the iron must be completely removed, or it is impossible to volatilize entirely the excess of arsenic; the button is then heated alone, as above mentioned, and the $(Co\ Ni)^4\ As$ weighed.

The cobalt is next slagged off, according to p. 456, until finally $Ni^4\ As$ alone remains. This compound is weighed and the amount of $Co^4\ As$ obtained by difference, according to the example on p. 459. When experience is wanting in regard to the proper temperature and the various phenomena of this assay, the resulting button may be again fused with a little borax on coal for a short time, and the melted glass examined, to see whether it is coloured brownish by oxide of nickel, or violet from a mixture of some cobalt remaining, or only blue from oxide of cobalt. In the latter case the button must be again weighed, and the amounts of cobalt and nickel reckoned anew, provided that all cobalt is now really separated.

When treating smaltite or cobalt speiss, which often contain so much arsenide of cobalt, that one fusion is not sufficient, the blast must be stopped when the borax-glass appears to be saturated in the first fusion, freed from the slag in water, and treated with fresh borax-glass, until all cobalt is removed, or the glass is again saturated. In the former case, that is, when apple-green spots appear on the surface of the solidified button, the slag is removed and the button weighed; in the other case, the cooled button is covered on the surface with a gray or dirty-yellow film of oxide, and the fusion must be repeated with a third and not too large portion of borax-glass, the operation being continued with fresh, but constantly decreasing portions of borax-glass, until all of the arsenide of cobalt is removed.

If the borax-glass, after saturation, were further treated with the blue flame, the arsenide of cobalt in the button would indeed gradually oxidize, but portions of it would be reduced again from the glass by the reducing action of the glowing coal, and the oxidizing process would only be prolonged. It is therefore better to remove the last of the cobalt with fresh borax-glass.

If the assay contains a notable amount of silver, this must be determined and deducted from the arsenide of nickel as sulphide of silver, in which state it occurs in the arsenide, before the nickel can be calculated. For this purpose the button is fused with enough test lead and some borax-glass on coal, and then cupelled according to p. 384. The resulting silver is reckoned as sulphide of silver, 100 parts of Ag yielding 115 parts Ag S.

When the substance is to be fused in a clay crucible instead of a coal crucible, the weighed amount is placed in it and freed from the excess of arsenic, if considerable, by gentle ignition, according to p. 435, until no more fumes are observed. To the mass, which is generally *sintered* together, three hundred milligr. of the fluxing and reducing agent are added, without mixing it with the substance, and then a cover of three spoonfuls of salt, as in the lead and bismuth assays. The fusion is conducted just as in those assays, and yields a perfectly fluid slag, through which may be seen the button of metallic arsenides at the bottom, when the coal cover is removed. After breaking open the crucible, the button is treated on coal according to p. 462, when the presence or absence of iron is at once ascertained, and the assay completed as there directed.

To determine bismuth at the same time, the substance, freed from excess of arsenic, is fused in the clay crucible with the flux and reducing agent, and an addition of iron and silver, as directed for the quantitative bismuth assay, p. 435. The combination of arsenides of iron, cobalt, and nickel, which has been more or less broken up in separating the bismuthiferous silver, either between paper on the anvil, or in the steel mortar, is fused on coal, or in a coal crucible, in the R. F., with soda and borax-glass, to a button, which is treated as before. The amount of iron being increased by the addition of the metallic iron, two or more portions of borax-glass are sometimes necessary to remove all the arsenide of iron from the arsenides of cobalt and nickel.

To show how accurately cobalt, nickel, and bismuth may be determined in a substance consisting mainly of metallic arsenides with an admixture of native bismuth, the following example is given :—

100 milligr. cloanthite (*Weissnickelkies*) from Schneeberg, pure and free from bismuth, was powdered and mixed in a clay crucible, with
15 „ metallic bismuth, also powdered,

and the excess of arsenic removed, according to p. 435.

To the nearly fused mass were added—

30 milligr. iron in one piece,
102 „ silver,
300 „ fluxing and reducing agent,
3 spoonfuls salt, as a cover.

The fusion yielded a button which showed on its surface a sharply-defined line, and could be so easily separated that the bismuthiferous silver was obtained almost pure. It was carefully melted with borax, according to p. 436, freed from slag, and found to weigh 117 milligr., and therefore contained $117-102 = 15$ milligr. bismuth.

The metallic arsenides and the little slag from the bismuthiferous silver button were fused with a small amount of soda in a coal crucible, the resulting button freed from arsenide of iron, as before directed, the excess of arsenic volatilized, and the button weighed. It weighed 46 milligr. Some of the nickel in cloanthite is generally replaced by cobalt, and the button was therefore treated with borax-glass, according to p. 456, by which 4 milligr. Co^4As were removed. The button therefore consisted of—

$46-4=42$ milligr. Ni^4As, with $\frac{60{\cdot}7 \times 42}{100} = 25{\cdot}5$ milligr. Ni,

And 4 „ Co^4As, with $\frac{61{\cdot}5 \times 4}{100} = 2{\cdot}3$ „ Co;

being about 28 milligr. nickel and cobalt together, which corresponds with the composition of cloanthite.

B. Assay for Cobalt and Nickel, and, if required, for Lead, Bismuth, or Copper at the same time, in Minerals, Ores, and Products containing Cobalt and Nickel, with perhaps other metals, combined partly with Arsenic and partly with Sulphur, or entirely with Sulphur, and containing at the same time a greater or less admixture of other metallic sulphides or earthy matters.

In this class are included, among minerals,—*cobaltite, glaucodot, danaite, cobaltic arsenopyrite, gersdorffite, amoibite, syepoorite, linnaeite, millerite, grunauite,* and *pentlandite;* further, all *cobalt* and *nickel ores* dressed on the large scale, generally mixed with earthy gangues and containing frequently mispickel or iron pyrites, and more rarely copper pyrites; among metallurgical products, *lead speisses,* which contain, besides arsenides of cobalt and nickel, much arsenide of iron and several metallic sulphides; also *niccoliferous* and *cobaltiferous lead matt* and *Rohstein.*

Dressed ores containing arsenides of iron, cobalt, and nickel, with only a greater or less amount of earthy ingredients, can be fused immediately in a clay crucible, according to p. 462; if, however, they are not free from an admixture of pyrites and mispickel, as well as sulphides of antimony, bismuth, and lead, which can be ascertained by a simple test on coal, they must, like the other sulphur compounds above named, be completely roasted on a clay capsule, according to p. 408; at first alone, but afterward, when they cease to evolve any odour, with fifty to sixty milligr. of carbonate of ammonia, which is triturated in the mortar with the roasted assay. The resulting oxide must be converted into arsenides by a large addition of metallic arsenic, which can be done in a clay crucible, or in a tube closed at one end.

When the conversion of the metallic oxides into metallic arsenides, or the "*arsenicizing,*" can be done outside of the laboratory, it is performed as follows: the roasted powder is mixed in the agate mortar with one hundred milligr. of pulverized metallic arsenic* and poured into the clay crucible, which is also to be used for the subsequent fusion. This is set on an iron wire ring in a square coal fixed in the coal-holder, as described for the fusion of the lead assay (p. 426); the crucible is then covered with a clay capsule, and the whole with a perforated coal cover. The coal-holder is kept at some distance from the lamp, and the hot products of combustion directed into the interior of the coal, when the crucible becomes s strongly heated that the arsenic begins to sublime and exerts a reducing action on the free metallic oxides and basic arsenates, forming arsenous acid and suboxide of arsenic, and at the same time changing into arsenides those metals which have a tendency to combine with it. The crucible is heated to low redness, so as to volatilize the excess of arsenic, as far as this is possible without access of air, and to *sinter*, or, under certain circumstances, fuse together, the metallic arsenides formed. When the excess of arsenic is expelled, the iron wire with the covered crucible is removed from the coal, as in the case of the melted lead assay, and placed on a stand to cool, with the cover still on.

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* A less quantity is not admissible, as the desired result might not then be attained, owing to the volatility of the arsenic.

When the arsenicizing must be performed in the laboratory, the mixture of ore, etc., with metallic arsenic is placed in the mixing-capsule and thence transferred to a perfectly dry glass tube, closed at one end, and gradually heated to glowing over the spirit-lamp. As before, the oxides and arsenates are converted into arsenides, while the excess of arsenic sublimes and condenses in the cool part of the tube. When cold the tube is cut with a file close under the sublimate, broken off, and the metallic arsenides, which generally form a dark, yellowish-gray powder, poured into the clay crucible destined for the fusion, while the tube is cleaned into it with the spatula and brush and the assay charged as will be directed below.

By either process the arsenicizing yields metallic arsenides, consisting, in presence of protoxides of nickel and cobalt, sesquioxide of iron, and oxides of copper, lead, and bismuth, of $Co^3 As$, $Ni^2 As$, $Cu^4 As$, and compounds of lead or bismuth with indefinite proportions of arsenic; the sesquioxide of iron is chiefly converted into $Fe^2 As$ and partly into proto-sesquioxide, while sulphate of lead is changed to sulphide. Any oxide of zinc remains unchanged, but is reduced by the subsequent fusion of the assay and volatilized during the treatment of the metallic arsenides on coal. If the roasted assay contained acids of antimony, they are reduced and changed to arsenides of antimony of uncertain composition: although these pass into the button of metallic arsenides formed during the fusion, they are volatilized while treating this on coal.

One centner of the finely-pulverized substance to be assayed having, if it contains metallic sulphides, been roasted thoroughly and the free oxides and basic arsenates thus formed converted into arsenides, the question arises, whether the substance contains lead or bismuth? If this is found to be the case by testing a little of the raw substance on coal, these metals can be separated during the fusion and quantitatively determined. It is only necessary, as directed in the bismuth assay, p. 435, to add to the arsenides in the crucible a piece of iron wire weighing about twenty milligr. and an exactly weighed amount of silver, from fifty to one hundred milligr., so that an alloy of bismuth or lead and silver is obtained during the fusion, from the weight of which the amount of lead or bismuth can be ascertained. If the substance contains none of these metals, or only an unimportant amount, which can be volatilized during the separation of the arsenide of iron and the excess of arsenic on coal (p. 462), the iron and silver are omitted, and the following substances at once added to the substance already in the crucible:

300 milligr. fluxing and reducing agent, which is pressed down somewhat with the spoon,
3 spoonfuls of salt as a cover.

The fusion is conducted as in the lead assay, p. 430, and the heat must be rather strong toward the end, so as to collect the arsenide to one button. After continuing the proper temperature for five or six minutes the arsenides collect in a round button at the bottom, while the earthy matters and the oxides which do not separate in the metallic state are completely slagged off. If iron and silver had been added to separate any lead or bismuth, the iron passes into the metallic arsenides, and the lead or bismuth forms an alloy with the silver, which joins with the arsenides to one button, but only forms an adherent part of it, and can easily be mechanically separated, as has been fully described in the bismuth assay, p. 435.

If the substance contains more nickel than cobalt, the metallic arsenides unite very easily to a button, but otherwise, the union is more difficult in proportion as the cobalt exceeds the nickel. A combination of arsenides of iron and cobalt, however, also fuses easily, and as these two arsenides can be separated without any important loss of cobalt, the evil mentioned can be remedied by combining the arsenide of cobalt with a corresponding amount of arsenide of iron. This is done by adding to the raw, or the roasted and arsenicized substance, according to the cobalt present, ten to twenty milligr. of iron, in case none had been added to separate any bismuth.

When the substance seems very poor in nickel and cobalt, so that it would be difficult to obtain a perfectly fused button, some collecting agent must be employed, which can be easily slagged off from the arsenides of cobalt and nickel. Arsenide of iron serves here also. Fifteen to twenty milligr. of arsenide of iron, formed directly in a clay crucible from iron filings and metallic arsenic, are added to the substance to be fused, or when the substance is arsenicized, ten to fifteen milligr. of iron filings are added also, in order that the necessary amount of arsenide of iron may be present already. The filings can be omitted when the roasted substance contains much oxide of iron, which secures the formation of arsenide, but a bit of iron and the necessary amount of silver must always be added when the lead or bismuth is to be determined.

When the fusion has been successfully accomplished, *i.e.* when the slag is perfectly fluid, and the melted button can be seen at the bottom, unless the slag contains too much protoxide of iron, or coal, which finally oxidizes at the expense of the carbonic acid of the alkalies, the crucible is set aside to cool on its stand; otherwise the assay must be kept at a melting heat for some time longer. When cool, the crucible is broken, and the button carefully separated from the slag on the anvil.

If silver has been added, the alloy is separated from the metallic arsenides between paper, or in the steel mortar, freed from adherent slag with borax (p. 437), and the lead or bismuth determined by the increase in weight. The arsenides and the slag last removed from the alloy are fused in the R. F. with soda, and the metallic arsenides separated, as given on p. 455. The substance is seldom free from nickel, and then the button entirely disappears when treated with fresh portions of borax-glass; if otherwise, there remains a button which can be recognized as arsenide of nickel, either by the signs given on p. 456, or by the last addition of borax, even when it only weighs one milligramme. If everything dissolves in the borax, the cobalt is to be calculated from the weight last noted down, according to p. 460; but if a little button of arsenide of nickel remains, it must be weighed, and the cobalt reckoned from the difference.

When the substance also contains copper, this will be present in the arsenical compound, which is then separated as follows: first, the arsenide of iron is removed with borax, according to p. 455; any zinc or antimony present is also volatilized at the same time; the excess of arsenic, with any remaining antimony, is volatilized next, and the arsenide weighed, after which the cobalt is removed, leaving a compound of arsenides of nickel and copper, which is fused with eighty to one hundred milligr. gold, the nickel separated with salt of phosphorus, according to p. 458, and the cupriferous gold weighed, after which the amounts of the metals can be found from the results of the respective weighings.

The wet way, although more complicated, may also be used for determining the copper and nickel after removing the cobalt. The button is dissolved in a test-glass with a mixture of about three parts dilute sulphuric acid and one part nitric acid, by warming it, and the copper and arsenic are precipitated with sulphuretted hydrogen.* The sulphides are separated by filtration from the solution containing all the nickel, washed with sulphuretted hydrogen-water, and the clear solution warmed in a porcelain vessel over the spirit-lamp until all the sulphuretted hydrogen is driven off. Some sulphide of arsenic generally separates, which must be removed by a second filtration, and then the protoxide of nickel is precipitated with not too concentrated a solution of potassa. The precipitate is collected and washed with hot water on a filter, dried, and ignited, or reduced to metallic nickel.

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* All operations where this gas is evolved should be performed without the laboratory.

In the former case the precipitate is transferred to the platinum capsule, and the ashes of the filter added, when the whole is strongly ignited and the amount of nickel deduced from the weight of the protoxide, after subtracting the weight of the filter-ash: 100 parts of Ni contain 78·38 parts Ni. The ash given by the filter must be previously determined; the protoxide of nickel should not follow the magnet.

In the second case the dry precipitate and the filter-ash are mixed with—

50 milligr. soda,
25 ,, borax-glass,

and a weighed gold button, and treated in a soda-paper cylinder on coal with the R. F., until all the nickel is reduced and combined with the gold: the amount of nickel is ascertained from the increase in weight of the gold button.

To determine the copper, the washed sulphides are dried and gradually heated in a deep porcelain vessel, over the spirit-lamp, so strongly that all the sulphide of arsenic volatilizes, and only sulphide of copper remains. To avoid loss, the dry precipitate is first transferred to the vessel and then the filter-ash added. The heat may be continued until most of the sulphide is converted into sulphate of copper, and when the odour of sulphurous acid ceases, six to eight times as much dry bisulphate of potassa is added, brought to fusion, and kept so at a moderate red heat until all the copper is dissolved. It is well to cover the vessel with a piece of platinum foil during this operation. The cooled mass is then dissolved by placing the vessel with its contents in another porcelain vessel half full of water and heating it well over the lamp, after which it is filtered, if necessary, the solution brought to boiling heat, and the copper precipitated by not too concentrated a solution of potassa. It is then collected on a filter, well washed with hot water, dried, ignited, and weighed with the filter-ash. After deducting the ash from the weight of the oxide of copper, the metallic copper can be easily ascertained by reckoning 79·8 parts of copper for every 100 parts of the oxide obtained. The result is more reliable when the oxide of copper is reduced to the metallic state with a gold button, just like the nickel above.

When one of the substances of the second class contains a notable amount of silver, this must be deducted, either as sulphide of silver from the arsenide of nickel, if the substance was fused in the raw state without addition of iron or silver, or as metallic silver from the reduced bismuth, if the substance was roasted, arsenicized, and fused with iron and silver, as directed in the bismuth assay (p. 436).

Example.—A lead speiss from the Freiberg Smelting-works, enriched by concentration for nickel and cobalt, but still very impure, was found to consist of arsenides of iron, nickel, and cobalt, and sulphide of copper, lead, zinc, and antimony; the trifling amount of silver was neglected. One ctr. being roasted, arsenicized, and fused with the flux and the addition of about twenty milligr. iron, and ninety milligr. silver, in a clay crucible, yielded a button, one-half of which was an alloy of silver and lead, while the other half consisted of metallic arsenides. Being detached in the steel mortar, and properly freed from slag, the alloy weighed 93·5 milligr.; and therefore the ninety milligr. silver had taken up 3·5 milligr. lead.

The arsenides and the slag, properly treated and freed from arsenide of iron and the excess of arsenic, yielded a combination of $m\,(Ni, Co)^4\,As + n\,Cu^6\,As$, which weighed 61·5 milligr. After removing the cobalt, the button weighed 48·5 milligr.; so that thirteen milligr. $Co^4\,As$ were removed, giving $\frac{61{\cdot}5 \times 13}{100} = 7{\cdot}99$ milligr. cobalt. If the speiss had been free from copper, the nickel could now have been also determined, but it was really necessary to fuse the button with a gold button weighing 85·6 milligr., when it was treated as directed on p. 458, and yielded a button weighing 93·9 milligr. The speiss, therefore, contained 8·3 milligr. copper. To determine the nickel, this copper was reckoned as $Cu^6 As$, and subtracted from the combination of arsenides of nickel and copper that had been found to weigh 48·5 milligr. The method of calculating the $Cu^6 As$ is as follows: 71·7 parts Cu and 28·3 parts As form 100 $Cu^6 As$; the 8·3 milligr. copper in the gold correspond, therefore, to $\frac{100 \times 8{\cdot}3}{71{\cdot}7} = 11{\cdot}57$ milligr. $Cu^6 As$, which being reckoned as 11·6 milligr. and subtracted from 48·5, leaves 36·9 milligr. $Ni^4 As$; or $\frac{60{\cdot}7 \times 36{\cdot}9}{100} =$ 22·4 milligr. nickel.

The speiss, therefore, contained

22·4	per cent.	nickel,
7·9	„	cobalt,
8·3	„	copper,
3·5	„	lead.

By making the assay according to the foregoing directions, when the necessary practice has been acquired, the whole examination can be made in about three hours, and the results obtained will agree very closely with those arrived at by the most carefully conducted wet method.

C. Assay for Cobalt and Nickel in Minerals, Ores, and Products containing Nickel and Cobalt in the oxidized state, combined with sulphuric, arsenic, or silicic acids, or other metallic oxides, and occasionally with water at the same time.

This class includes, among minerals and ores, *bieberite, erythrite, earthy cobalt bloom, lavendulan, morenosite, emerald nickel, annabergite, röttisite genthite, pimelite, earthy cobalt,* and *cobalt* and *nickel ores roasted* on a large scale; among products, *speisses roasted* on a large scale, *smalt, cobaltiferous* and *niccoliferous slags* which fall from refining the slags and tallic arsenic are mixed thoroughly with it. If the oxide consists chiefly of protoxide of nickel, or a mixture of protoxide of nickel and oxides of cobalt, in which the former prevails, the resulting arsenides can easily be melted to one button during the fusion in the crucible; if, however, oxide of cobalt prevails, the resulting arsenides melt with difficulty, and about fifteen milligr. of iron filings must be added, so as to form arsenide of iron, which produces a fusible combination with the arsenide of cobalt in the subsequent fusion. The arsenicizing is performed just as described on p. 467, and if a stronger heat than necessary was employed toward the end, the metallic arsenides, if easily fusible, may be melted to a button. After cooling in the covered crucible, the arsenides are charged with three hundred milligr. flux and reducing agent, which is poured upon them, and then covered with three heaped spoonfuls of salt, after which they are fused according to p. 468.

Another method of arsenicizing, with which the fusion of the metallic arsenides formed is at the same time combined, consists, according to Fritzsche, in treating fifty milligr. of the oxides, mixed with one hundred milligr. arsenate of potassa, and thirty milligr. borax-glass in a soda-paper cylinder in a coal crucible with the R. F., until the arsenides formed by the liberated arsenic have united to a button, and the carbonate of potassa produced has sunk with the borax into the coal. This should be done outside of the laboratory. Small admixtures of metallic oxides, which are neither converted into arsenides nor volatilized, remain in a divided state on the crucible without exerting any injurious effect on the union of the metallic arsenides.

When the arsenicizing must be done in the laboratory another method should be adopted, so that the fumes of arsenic cannot escape. Fifty milligr. of the oxides mixed with one hundred milligr. powdered metallic arsenic are placed in a glass tube closed at one end, and heated, as described for ores, etc. (p. 467) over the spirit-lamp, until the sublimate of arsenic ceases to increase; or, to avoid all mechanical loss, the mixture is inclosed in a soda-paper cylinder, made of a strip of fine filter-paper forty-five millim. long and twenty millim. wide, as in the silver assay, and placed in a tube eighty to ninety millim. long and about ten millim. wide, closed at one end, as shown in Fig. 76, *a*. A roll of ordinary filter-paper is then inserted as far as *b*, to absorb the moisture evolved from the charred soda-paper, and the assay is gradually heated in the spirit-flame to redness, while the tube is occasionally turned to prevent the charring paper from adhering to the glass. It is kept in this condition until the sublimate, *c*, ceases to increase.* By carefully heating it at first, the paper is charred without opening in any part, or adhering to the glass, and therefore prevents all mechanical loss.

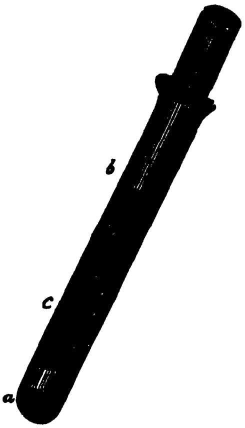

Fig 76.

If the mixture has been placed at once in the tube, the lower part of the tube is cut off after the arsenides have been formed, and these are poured into the mixing capsule and mixed with—

50 milligr. soda, and
20 ,, borax-glass.

This is then transferred to a soda-paper cylinder and fused in a coal crucible in the R. F. If, however, the mixture to be arsenicized had been wrapped in soda-paper, the glass tube is carefully inverted and the assay shaken at once into a cavity in coal, or a coal crucible, wide enough for the fusion. Sometimes the charred paper will adhere to the glass, if it has been soaked with too much soda and heated too strongly, and it is then necessary to detach it with a wire or splinter of wood.

50 milligr. soda and
20 ,, borax-glass

are then put on the charred cylinder and the whole fused in the R. F., with only a feeble flame at first, directed upon the flux alone, and afterward directed as much as possible against the bottom of the cavity, between the assay and the coal, so that the arsenides may be fused from below upward, while the envelope is consuming.

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* The sublimate adhering firmly to the tube is best detached, after taking out the assay, by simply pushing through the tube another tube open at both ends.

While the arsenides, formed in either way, are fusing to a button, arsenic still volatilizes, until the remaining compound corresponds nearly or quite to the composition $(Ni, Co)^4 As$, provided there is no iron present; the fluxes either sink into the coal or combine to a transparent slag, according as the oxides assayed are free from or contain other metallic oxides. When the fusion is performed in a coal crucible, it is well to add half a spoonful of neutral oxalate of potassa and treat the assay further with the R. F., before removing the metallic button, as this causes all the slag to sink into the coal, leaving the button quite free. With the aid of a glass it may easily be seen whether any little buttons are scattered about, and if any one is found, which is seldom the case, it is detached with the point of the knife, rolled up to the main button, and at once fused with it in the R. F.

In separating the arsenides now formed, the first question is, again, whether the button contains arsenide of iron or not? The manner in which this is ascertained and the iron separated is given on p. 462. The nickel and cobalt are then also determined by the process already described; but in calculating their amounts it must be borne in mind that only fifty milligr. were taken for the assay.

100 parts cobalt yield 126·66 parts of protoxide, and
100 „ nickel „ 127·58 „ „ „

so that the amount of these oxides can be easily calculated after determining the respective metals; or they may be calculated directly from the following data:

$$100 \text{ parts } Co^4 As = 61{\cdot}5 \text{ cobalt} = 77{\cdot}9 \; Co\,O,$$
$$100 \;\;,,\;\; Ni^4 As = 60{\cdot}7 \text{ nickel} = 77{\cdot}4 \; Ni\,O.$$

The methods heretofore described may be advantageously used in the analysis of niccoliferous and cobaltiferous substances. The other constituents of the assay having been separated by the known methods and the oxides of cobalt and nickel precipitated together, they are washed, ignited, and weighed. After being then pulverized and again ignited, fifty milligr.

of them are arsenicized, which should be done in a clay crucible if possible, because then there can scarcely be any mechanical loss; and after being fused to a button they are separated, according to p. 455 *et seq.*, and the percentage of cobalt and nickel or their oxides thus ascertained.

Example.—Suppose that from 1 gramme of nickel speiss a precipitate of the two oxides had been obtained, which weighed, after ignition, 0·475 grm., and from 50 milligr. of these oxides 64 milligr. of $(Ni, Co)^4 As$ were produced; the cobalt being separated, there remained 43·8 milligr. $Ni^4 As$; then the 0·475 grm. of metallic oxides would yield—

$$1{\cdot}50 : 43{\cdot}8 :: 0{\cdot}475 : x \text{ grm. } Ni^4 As;$$

$$x = \frac{43{\cdot}8 \times 0{\cdot}475}{50} = 0{\cdot}4161 \text{ grm. } Ni^4 As, \text{ or}$$

$$\frac{0{\cdot}4161 \times 60{\cdot}7}{100} = 0{\cdot}2525 \text{ grm. } Ni; \text{ and the}$$

speiss, therefore, contained 25·25 per cent. nickel.

2. Since $64 - 43{\cdot}8 = 20{\cdot}2$ milligr. $Co^4 As$ were obtained, the amount of arsenide of cobalt would be—

$$50 : 20{\cdot}2 :: 0{\cdot}475 : x \text{ grm. } Co^4 As;$$

$$x = \frac{20{\cdot}2 \times 0{\cdot}475}{50} = 0{\cdot}1919 \text{ grm. } Co^4 As, \text{ or}$$

$$\frac{0{\cdot}1919 \times 61{\cdot}5}{100} = 0{\cdot}118 \text{ grm. } Co; \text{ and the speiss,}$$

therefore, contained 11·8 per cent. Co, and 37·05 per cent. of both metals together.

E. Assay of Minerals and Products consisting of alloys, or arsenides and sulphides, in which there is more Copper than Nickel.

This class includes, among minerals, *breithauptite*; among metallurgical products, *niccoliferous* and *cobaltiferous black copper*, similar *copper matts* and *slags*, which are rich in suboxide of copper; among artifical products, *German silver*, or *packfong*, *tutenag*, and similar alloys.

These substances cannot be quantitatively examined for cobalt and nickel according to the method given under *A* to *D*, since they contain too much antimony and copper.

In case of alloys of nickel and cobalt with antimony, one hundred milligr. are dissolved in nitric acid, the solution filtered from the residue of oxide of antimony, and the oxides of cobalt and nickel precipitated with potassa, collected on a filter, washed, dried, ignited in the platinum capsule, and the ignited oxide, which may contain other metallic oxides with a little antimonic acid and oxide of antimony, arsenicized according to one of the methods on p. 476. They are then fused and treated for cobalt and nickel, which are determined according to p. 477 *et seq.*

Products containing more copper than nickel must likewise be dissolved in nitric acid, or if necessary in aqua regia. When the amount of nickel and cobalt is supposed to be very small, more than one hundred milligr. are taken, and slags must be very finely pulverized. From the solution, copper, lead, antimony, etc., are precipitated by sulphuretted hydrogen, the solution filtered, and the precipitate washed with sulphuretted hydrogen water, which is added to the solution, and the whole evaporated until it has no odour of that gas; it is then heated to boiling, any protoxide of iron present converted into sesquioxide by adding a little nitric acid, and the metallic oxides precipitated with potassa. If the solution of the metallic oxide in aqua regia contained arsenic acid, this is partially precipitated in combination with protoxide of nickel and cobalt, but does no harm. The precipitated oxides are collected on a filter, washed with hot water, dried, ignited, and arsenicized, and the resulting arsenides fused and treated for cobalt and nickel.

If the copper, lead, etc., are to be determined, the precipitated sulphides are dried and gradually heated to redness in a thin porcelain capsule, so as to expel the excess of sulphur, sulphide of arsenic, and most of the antimony, after which they are fused with bisulphate of potassa in the same capsule. When the resulting sulphates are treated with hot water, the sulphate of lead remains behind, but the sulphate of copper goes into solution, and, after filtration, can be precipitated as oxide with potassa. If pure, the sulphate of lead and the oxide of copper may then be dried, ignited, and weighed; or they may be determined by reduction assays; for copper, according to p. 414, and for lead, according to p. 432.

8. THE IRON ASSAY.

The iron-smelter has sufficient means for ascertaining the amount of metal contained in his ores by melting them, with proper fluxes, at a high temperature, in fire-clay crucibles lined with charcoal, as well as by the simpler modes of treating them in the wet way; for example, by the methods of Fuchs, Marguerite, and others. As it might be desired to determine the iron in an ore when the necessary apparatus for the latter methods was not at hand, and as it is, moreover, possible to determine the predominating earthy constituents or admixture of an iron ore, approximately and accurately enough for practice with the blowpipe and wet way combined, this method of proceeding will be described here.

The different iron ores which in general are smelted on a large scale may be distinguished as follows:

1. *Magnetite,* proto-sesquioxide of iron, but often mixed with different silicates, as well as sulphides.

2. *Specular iron* and *hematite.* Both consist principally of sesquioxide of iron, but are seldom free from oxide of manganese and admixtures of earthy substances, as hornblende, chlorite, quartz, ferruginous quartz, hornstone, feldspar, barytes, etc.

3. *Limonite, yellow ochre,* and the related *bog iron, ores.* These consist of hydrated oxide of iron, with more or less oxide of manganese and phosphoric acid, and often with the same admixture of earthy substances as hematite.

4. *Clay iron ore,* which is to be regarded principally as a mixture of hydrated oxide of iron with alumina and sand.

5. *Silicious iron ore,* consisting principally of silicate of the protoxide or sesquioxide of iron in different proportions, but seldom free from earthy silicates.

6. *Siderite* (*spathic iron*), consisting, when pure, of carbonate of iron, but often mixed or combined with carbonates of manganese, lime, and magnesia, and, as *black band,* with coal and clay-slate.

7. *Spherosiderite.* This contains the iron combined, it is true, with carbonic acid, like siderite, but it is distinguished

from the latter by foreign admixtures, such as clay, which is so intimately united with the carbonate of iron that the ore possesses a homogeneous appearance. Phosphate of iron, phosphate of lime, and pyrites are also present as admixtures.

It will be seen from the above that most ores contain, besides iron, more or less of other constituents or admixtures, which must be of interest to the iron metallurgist, when, in determining the iron in an ore, the earthy parts, as silica, alumina, lime, etc., which have an important influence upon the slag formation, are to be also approximately determined.

PREPARATION OF THE ASSAY POWDER.

A sufficient quantity of the ore to be examined must be pulverized, a larger portion being taken if the admixture of foreign substances in the ore is large, so that a proper average may be obtained. The ore is first broken up with the hammer and then in a mortar. For this, an iron mortar can be used, in which the ore is pounded, but not rubbed. When the assay powder is rather fine, it must be carefully mixed, and small quantities taken from different parts with the ivory spoon. The selected portions are then to be very finely powdered in an agate mortar, and dried in a porcelain dish over a lamp at about 100° C.

MODE OF CONDUCTING THE ASSAY.

The ores described above are of such a nature that the oxides of iron contained in them may be either entirely dissolved out by acids, provided they are heated, or else a residue is left, which contains more or less iron. In the first case, if a quantitative determination of the earthy parts is not necessary, no fusion is required; in the latter case, however, the residue must be first decomposed by a fusion with the proper fluxes on charcoal before the blowpipe. The fusion is made as follows :—

One ctr. of the prepared assay powder is weighed out, and put into a test-glass of sufficient capacity, and enough hydrochloric acid for its solution gradually poured over it.

If the ore consists principally of carbonates, the acid must be added in very small portions, in order to prevent a violent effervescence. If upon the addition of a little acid it appears to be free from the former, the necessary amount of acid may be immediately added. If the ore does not dissolve at the ordinary temperature, it must be digested with the acid over the spirit-lamp, either until all is in solution, which, however, only occurs with perfectly pure ores in which the oxides of iron are not combined with silica, or else until nothing more dissolves. A few drops of nitric acid are then added to the boiling-hot solution, and heat again applied. The solution is at first coloured dark, if it contains protochloride of iron, and then suddenly becomes pale yellow, as the protochloride is changed to the sesquichloride.

It is then diluted with distilled water, and the residue, if there is any, brought upon a filter, and washed well with hot water. When the residue is perfectly white, it need not be treated any further, if the iron alone is to be determined, because it is then usually either quartz or earthy silicates, seldom mixed with barytes, and entirely or nearly free from iron. The iron may then be immediately precipitated as described below. If, however, the residue is not perfectly white, but has a yellowish, red, or gray colour, the filter must be spread out, and dried in a porcelain dish over the lamp. As soon as it is dry, it can be mixed on the filter with three parts, by volume, of soda, and one part borax-glsss, wrapped up in the filter-paper, and laid either in a cylindrical hollow bored in the cross section of a good piece of charcoal, or in a charcoal crucible, and the whole mass melted with the O. F. to a clear, transparent bead. This, when cold, must be taken from the charcoal, pulverized in the steel mortar, and the powder treated at once in a porcelain dish over the lamp with dilute aqua regia, and evaporated to dryness, or it may first be digested with dilute hydrochloric acid, nitric acid being added to the solution afterwards. If the ore contained barytes or other sulphates which were not dissolved by the acids, a little sulphide of sodium is sometimes formed during the fusion on charcoal from the decomposition of the sulphates. It is better in this case to use at first only dilute hydrochloric acid, so that the sulphur of the sulphide of sodium may be driven off as gas combined with hydrogen, and to add afterward the nitric acid necessary for the oxidation. The dry mass is then moistened with a few drops of hydrochloric acid, the soluble salts dissolved out with hot water, and the silica, which remains unaltered, is collected on a filter and well washed. The filtrate and the wash-water are then added to the first solution, from the direct treatment of the ore with acid, and after it has been ascertained by the addition of a few drops of sulphuric acid that no barytes is present, the sesquioxide of iron and alumina are precipitated with ammonia. If, however, baryta was present in the solution, the resulting sulphate of baryta must be allowed to settle from the warm solution and separated by filtering.

The precipitate of sesquioxide of iron and alumina is filtered off, well washed, and the filter laid upon several thicknesses of filter-paper and unfolded. As soon as the greater part of the water has been absorbed by the paper, the precipitate must be carefully taken from the filter with a knife and put into a porcelain dish. The filter, which still holds traces of the precipitate, is moistened in a porcelain dish with some hydrochloric acid, washed several times with a little water, and this solution added to the main precipitate. This being done, a concentrated solution of caustic potassa is added and the alumina dissolved out over a spirit-lamp. During this operation the sesquioxide of iron decreases considerably in bulk. After the whole mass has been boiled feebly for a short time, it is diluted with water, and the sesquioxide of iron collected on a filter, well washed, and laid upon porous paper so that the greater part of the water may be absorbed. As soon as the filter is tolerably dry, it is unfolded and dried thoroughly, with the sesquioxide of iron still upon it, in a porcelain dish over a lamp.

When the filter is dry, the sesquioxide of iron is put for the present in a little porcelain cup, and the filter is burned thoroughly in a platinum capsule, to which the sesquioxide of iron is then added and the whole gradually heated to redness.

The sesquioxide of iron must be separated from the filter, because the carbonic oxide gas produced by the gradual burning of the latter, when the filter is being reduced to ashes, is apt to act upon sesquioxide of iron when heated to redness, and if the precipitate had not been removed before burning, a part of it would be reduced by the carbonic oxide to protoxide of iron, thereby giving too low a percentage of iron. The dry sesquioxide of iron must also be carefully heated to redness, for if heated too strongly at first, the compact oxide would decrepitate violently, owing to a small amount of water still present, and a mechanical loss would ensue. It is, therefore, better to cover the platinum capsule at the beginning of the operation with a piece of foil.

The ignition being finished, the precipitate is weighed, the weight of the filter-ash deducted and the amount of iron calculated from the weight of the sesquioxide. One hundred parts of the sesquioxide of iron correspond almost exactly to seventy parts of metallic iron, so that it may be conveniently calculated as seventy parts.

If the percentage of raw iron which the dry assay in a charcoal crucible would give is required, it may be easily calculated by assuming the raw iron from the crucible to contain on an average in one hundred parts, ninety-six parts of iron and four parts of carbon.

Assuming that 50·8 milligr. sesquioxide of iron were obtained from an iron ore, this would give—

$$\frac{50{\cdot}8 \times 70}{100} = 35{\cdot}56 \text{ milligr., or per cent. iron, or,}$$

$$\frac{100 \times 35{\cdot}56}{96} = 37 \text{ per cent. raw iron.}$$

SIMULTANEOUS QUANTITATIVE DETERMINATION OF THE PREDOMINATING EARTHY CONSTITUENTS.

If it is required at the same time to determine approximately the other constituents of an iron ore, which may be present in considerable quantity, *e. g.*, silica, alumina, and lime, and possibly magnesia and manganese, the process is as follows :—

a. To ascertain the percentage of *silica*, the residue from the digestion of the ore with acids must be fused on charcoal with soda and borax-glass, the melted bead pulverized in the steel mortar, and then treated with dilute aqua regia; but if it contains barytes, it must be treated with dilute hydrochloric acid and afterward, if protochloride of iron is present, with nitric acid. It is then evaporated to dryness, the dry mass moistened with hydrochloric acid and dissolved in hot water. The residue of silica is collected on a filter, thoroughly washed and dried with the filter in a porcelain dish. The filter is then carefully folded so that the silica remains in the centre, and incinerated in a platinum capsule, covered at first, over a spirit-lamp, until the silica appears perfectly white. After weighing the silica and ash, the weight of the latter is deducted and the percentage of silica calculated. The silica must be as dry as possible before ignition, as the presence of a little water is apt to occasion a mechanical loss of small particles of finely-divided silica, which are carried off by the sudden development of steam.

b. To determine the *alumina,* carbonate of ammonia is added to the filtrate from the silica, if it is free from baryta or iron, and also to the solution resulting from the treatment of the sesquioxide of iron with potassa, after the same has been slightly acidulated with hydrochloric acid. The precipitated alumina is then collected on a filter, washed, dried, ignited and weighed. The weight of the alumina is found by subtracting the ash of the filter.

c. The *lime* is precipitated by oxalic acid from the ammonical filtrate from the sesquioxide of alumina and iron, and the precipitated oxalate of lime, after being filtered, washed, and dried, is converted into carbonate of lime by heating to low redness. The amount of lime is calculated from the weight of the former, after deducting the filter-ash. One hundred parts of carbonate of lime contain 56·1 of lime.

d. Finally, the *magnesia* and *protoxide of manganese* are precipitated together in the filtrate from the oxalate of lime by a solution of salt of phosphorus. The precipitate is seldom large enough to be determined accurately, but after filtering it can be tested B. B. for manganese according to p. 193 *et seq.* If the ore is rich in manganese, it is better to precipitate the latter with sulphide of ammonium from the ammonical solution, and then the lime and magnesia with the proper reagents, as has been described under lime. In this case traces of alumina which remain in the solution will also be precipitated.

If the ore contained barytes, a few drops of sulphuric acid added to the filtrate from the silica will prove the presence of baryta; for the fusion of the earthy residue with soda and borax-glass decomposes the barytes, and the baryta is brought into solution when hydrochloric acid is added to the melted assay, to drive off the sulphur from the sulphide of sodium.

Small quantities of sulphur and arsenic and of sulphuric, phosphoric, and arsenic acids, can be easily found as described in their respective places, under qualitative examinations, but they cannot be determined quantitatively. Such determinations can only be made with larger quantities by regular analysis, and for this purpose works on analytical chemistry must be consulted.

9. THE CHROMIUM ASSAY.

The quantitative determination of chromium with the blowpipe, as described by Hilgard, is, in general terms, as follows: The substance to be assayed is decomposed by fusion with nitre, the melted mass dissolved in water, the solution evaporated to dryness, and the chromate of potassa converted, by fusion with bisulphate of potassa, into a constant compound of sulphate of potassa with sesquioxide of chromium, $\dot{\mathrm{K}}\,\dddot{\mathrm{S}} + \dddot{\cancel{\mathrm{C}}}\mathrm{r}$, from which the percentage of chromium in the substance can be easily calculated.

A. Fusion with Nitre.

a. When the substance does not contain either lead, bismuth, or cadmium.

In this class belong the minerals, *chromite*, *chrome-ochre*, and the similar silicates containing chromium (*vide* alumina), also the artificial product *chrome-green*, which, however, is often mixed with various substances. Chromite must be elutriated, for otherwise a perfect decomposition will not take place. This preparation is not necessary for chrome-ochre and chrome-green, as they are both easily decomposed by nitre.

The weighed quantity, of chromite one hundred milligr., but of chrome-green only fifty to seventy-five milligr., is put into a small platinum crucible of the same size as the clay crucible used in quantitative assays, and three heaped spoonfuls of nitre are added; with chrome-green, however, the latter must be added gradually, on account of the violent oxidation which takes place. The crucible, covered with a small platinum dish of the size and shape of the roasting capsule, is placed in a triangle of platinum wire, which is fastened to the ignition-ring. The fusion is made over the flame of an ordinary spirit-lamp, provided with a sheet-iron chimney. This gives in most cases sufficient heat, provided the bottom of the crucible is placed in the apex of the flame, where the temperature is highest. The fusion must, however, be carefully conducted, so that any violent bubbling of the mass or spurting may be prevented. The creeping up of the nitre on the sides of the crucible to the triangle, and the swelling of the melted mass, cannot always be avoided, so that care must be taken to bring everything into the solution. With chromite, the evolution of gas lasts from twenty-five to thirty minutes, and then the mass becomes quiet, the ore being entirely decomposed. It is advisable, however, always to test the residue for chromium.

b. When lead, bismuth, tin, or cadmium is present in the substance.

Under this head belong the minerals *crocoite*, *phœnicochroite*, and *vauquelinite*; among artificial products, the chrome colours, chrome-yellow, chrome-red, and chrome-green, when the latter is mixed with carbonate of lead.

Of the above-mentioned substances, one ctr. is intimately mixed in an agate mortar with three to four spoonfuls of fine silica and two spoonfuls of nitre. The fusion is made as in *a*, except that one spoonful only of the mixture is put into the crucible at a time, the pestle and mortar being cleaned by rubbing them with two or three half-spoonfuls of nitre. The fluid mass must be perfectly clear at the end of the fusion, small flakes of silica being visible upon the sides and bottom of the crucible. Undecomposed portions of the chromium compound appear dark red and become orange-yellow upon cooling. Should this be the case, it is partly owing to an imperfect mixture of the mass, and partly to an insufficient quantity of silica; such an assay should be repeated.

The decomposition of substances of this class requires from five to ten minutes. If at the end of the fusion the mass appears dry, a spoonful of nitre may be added.

B. Further treatment of the fused mass.

The fused mass is next dissolved in water, in which operation it must be noticed whether manganese or silica is present. The former is separated from the solution by a few drops of alcohol, while the latter is precipitated more quickly by boiling. The crucible and its appurtenances are put into a dish, which is used afterward for the evaporation, and boiled with water for not longer than half an hour, when the solution is poured into a beaker glass, the crucible being put with it. If, however, antimony is present in the substance, which is seldom the case, and if the solution must not be heated, the crucible is allowed to become perfectly cool, and is then put into a small beaker glass containing about a quarter of an inch of water, and allowed to remain until all is dissolved. The solution is then poured into a test tube, a spoonful of nitrate of ammonia added, and the whole shaken well, the mouth of the tube being closed with the thumb. The contents are afterward put back into the beaker glass, the tube and thumb being carefully rinsed off into it.

The solution, obtained in either of the above-mentioned ways, is then filtered and the evaporation commenced as soon as the greater part has passed through the filter. This is performed in the same vessel that is used afterward for the fusion with the bisulphate of potassa; it is made of platinum, and is of the same shape as the larger porcelain dish. During the evaporation the dish is covered with a closely-fitting glass bell, which can be made by cracking off the upper half of a white vial. The dish is placed on the support over a spirit-lamp, the neck of the glass bell being secured in a clamp which is fastened to the blowpipe lamp. By this arrangement the fluid can be well boiled without any danger of loss. During the evaporation the filtering and washing may be completed, and the filtrate carefully poured from time to time into the dish, without soiling the neck of the glass cover, the lamp being always previously withdrawn.

As the salt is apt to effloresce during the evaporation, it is advisable to commence the next operation and to prevent any swelling of the mass by adding sulphuric acid and bisulphate of potassa. Concentrated sulphuric acid is dropped in, until the yellow solution changes to orange-colour, when four or five drops more may be added. The amount of bisulphate of potassa varies with the amount of nitre used; three to six or seven heaped spoonfuls being the average quantity, although an excess does no harm.

The last part of the wash-water is not added until copious vapours of binoxide of nitrogen and sulphuric acid begin to escape, when the glass bell and any portions of the mass that may have swelled up on the edge of the dish are rinsed off with the wash-water, and the glass finally cleaned with a few drops of distilled water. The dish, however, must not be more than one-third full. The evaporation is now carefully completed, keeping the dish covered by a piece of platinum of the shape of a watch-glass. Toward the end of the operation the mass begins to expand slightly, the last portions of water go off, and soon sulphuric acid is set free. The salt then forms a thick, green, syrupy fluid, which turns peach-blossom-colour when further heated, and yields a double salt of chromium and potassa.

After the dense fumes cease, the lamp is removed, the cover quickly taken off, laid in a porcelain dish, and the platinum dish seized by the edge and tipped and turned round and round, while the mass is still fluid, so that the latter may be spread out as much as possible. When the salt is dry, the dish is put back upon the support, six to eight drops of concentrated sulphuric acid are added and the whole covered up and heated again. If peach-blossom-coloured particles are still visible in the green mass when cool, too little bisulphate of potassa has been used; more must now be added and the whole again heated with a few drops of sulphuric acid. In any case, the residue must be so soft that it can be quickly removed from the dish with a spatula or glass rod. Generally there results a tough, green syrup, very soluble; sometimes, however, a soft, but dense crystalline mass. If the latter has become too hard, it requires too much time for its solution in water without heat; if, on the contrary, it is too soft, or perhaps still fluid, it would be heated too much by the contact with water, and there would be danger of an alteration in the composition of the salt. The first difficulty may be remedied by the addition of a few drops of sulphuric acid and heating; the latter by driving off the excess of acid.

The resulting salt, when of proper consistency, is stirred up and well disintegrated with a platinum spatula, and enough water added at once to prevent any spontaneous heating. As soon as everything is dissolved, aided by stirring and pressing, the solution and residue are poured back into the beaker glass first used, the cover rinsed off, and any adhering particles washed off with a little of the solution from the dish, which is finally cleaned with the forefinger and a few drops of water. If the inside of the platinum dish is not perfectly smooth, small portions of the chromium salt are apt to adhere closely, and they must be removed by a fusion with nitre.

About one-third of a spoonful of powdered protochloride of mercury is then added to the cloudy solution and quickly dissolved by stirring, and a little very dilute sulphide of ammonium is then slowly dropped in. Upon the addition of each drop, the solution must be stirred and carefully watched to see if the flakes of chlorosulphide of mercury float in a perfectly clear fluid. These flakes are intended to collect and inclose the pulverulent chromium salt. The solution generally becomes clear when the flakes assume an olive colour, and there is only a slight excess of the chloride of mercury. The residue must be thoroughly stirred up, for if any of the chromium salt remains upon the bottom of the glass, it will make the solution cloudy again.

The flakes of chlorosulphide of mercury generally settle quickly, and the filtration can be immediately commenced. Swedish filter-paper, cut one and one-half inch in diameter, or filter-paper previously treated with acid, is recommended. When the main part of the solution has passed through, the precipitate is brought immediately upon the filter, and a little chloride of mercury and water added to it. This must also be done before each fresh addition of the wash-water. The washing is finished in about thirty to forty-five minutes, the wash-water being tested with chloride of barium, or upon platinum foil. Moreover, the precipitate should be spread out as much as possible, and not collected in the lower part of the filter.

The filter is then carefully removed from the funnel, dried between blotting-paper, and finally cautiously pressed between the folds of the latter into the form of a cube.

This is incinerated over a spirit-lamp in a platinum capsule, most of the mercury salt being driven off at the same time. A square coal is then prepared as in roasting copper ores, the dish placed upon the wire, and the whole treated with the blowpipe until completely incinerated. It is advisable during this operation to turn the mass over. The dish is now taken out of the coal, the screen of the coal-holder closed, and the ignited mass put into the platinum crucible, which has been previously weighed with its little platinum tripod. The crucible, placed in the coal, is covered with the capsule, and a charcoal cover is placed over the whole. It is now heated to the utmost for five or six minutes. A very thin crucible may be used for this operation, in order to prevent any overloading of the balance, but a thicker one is needed for the fusion.

After the heating, the precipitate appears of a fine green colour throughout, with the exception sometimes of a few half-fused particles. If not heated sufficiently, it is better to repeat the operation, having added a small piece of carbonate of ammonia. The crucible is finally taken out, placed upon the triangle, and a few drops of concentrated sulphide of ammonium poured upon the porous mass. The whole is then carefully dried over the spirit-lamp and finally heated to redness, to drive off all the sulphur.

Any chromate, which might have been formed during the first heating, will be decomposed by the last manipulation, and the resulting compound can be regarded as sulphate of potassa with sesquioxide of chromium, $\mathrm{K}\,\mathrm{S}+\bar{\mathrm{C}}\mathrm{r}$, containing in 100 parts 47·3 sesquioxide of chromium. This result can also be proved by washing the precipitate upon a filter, until the whole of the sulphate of potassa is dissolved. The remaining sesquioxide of chromium is then ignited and weighed, when it will generally agree with the first result, giving perhaps a very little more.

Although this assay may appear to be very tedious, it can be completed in one and a half to two hours.

The author finally presents a few modifications of the assay, which are necessary in the presence of certain substances.

It sometimes occurs that the solution from the first filtration becomes cloudy when diluted; this is owing to tin or bismuth, and shows that there was not sufficient silica added for the fusion. The same may occur in the presence of antimony if some nitre is not put on the filter.

In the presence of metallic acids, it is generally advisable to acidify the solution strongly with a few drops of sulphuric acid and wash somewhat longer, especially when phosphoric acid is present. Tungstic acid is precipitated in the acid solution, and is apt to occasion a bumping of the fluid during the evaporation. During the solution of the fused mass of sulphate, however, it prevents the distintegration of the residue, which separates quickly in flocculent particles, thus making the employment of chloride of mercury unnecessary. The tungstic acid is finally removed by digesting the chromium salt with ammonia on the filter.

In presence of antimony, the solution must not be boiled after the fusion, but must be shaken up in a test tube with nitrate of ammonia, in order to render the silica flocculent. The washing must be done with water containing a little nitre in solution. In dissolving the bisulphate, one or two spoonfuls of tartaric acid are put into the beaker glass, the solution kept strongly acid, and finally well washed with chloride of mercury.

If magnesia is present in large quantities, it is best to add silica for the fusion. When the ore contains copper, and no silica has been added, yellow flakes of sesquioxide of chromium and oxide of copper will appear during the solution of the mass. The solution must then be boiled with a little caustic potassa, when the yellow flakes contract to small black scales, containing no sesquioxide of chromium.

10. EXAMINATION OF COALS.

The usefulness of the blowpipe has been increased by employing it in the examination of coals, which both Chapman and Kerl have described. This examination includes the most important determinations that are usually made by analytical methods, viz., the hygroscopic moisture, the coke production, the amount of ash, and the absolute heating power.

a. Determination of the moisture.—From one hundred and fifty to two hundred milligr. coarsely pulverized coal are put into a porcelain dish and the latter placed upon the support of the blowpipe lamp. It is then heated moderately, either over the oil or spirit-lamp, for at least five minutes. A piece of white filter-paper laid in the dish must, however, show no signs of charring. Upon cooling, the assay is weighed and the result controlled by a second heating. As many kinds of anthracite decrepitate upon being heated, the dish should be covered with a small watch-glass. The percentage of moisture is lowest in anthracite; in bituminous coals it is usually three to four per cent., seldom six to seven, and reaches its maximum in lignite and brown coals, which contain twenty per cent., and sometimes more.

b. Determination of the coke production.—An ordinary clay crucible may be used for this operation, or the platinum capsule recommended by Chapman. The latter consists of two capsules, upon one of which there is an arm to prevent its falling over (Fig. 77). The edge of the upper one is bent in a little, so that it serves as a cover for the other, both being made of very thin platinum foil, in order that their weight may not exceed two grammes and to permit of their being used upon the blowpipe balance. The amount used for an assay is one hundred and fifty to two hundred milligr. The heating of the platinum or clay crucible, the latter being covered with a roasting capsule, is best accomplished in a square coal arranged in the holder as in the assay of ores. The caking is completed after the substance has been heated to redness for four or five minutes. When a clay crucible is used, the coke must be taken out, which can be done with perfect ease.

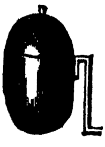
Fig. 77.

The coke has sometimes a fused, porous appearance and metallic lustre, as from caking or fat coals; sometimes it is only sintered together and partially fused, as from most of the free-burning or non-caking coals; sometimes not at all fused, but pulverulent and sandy, when obtained from anthracite, the most of the close-burning coals, and lignite. The weight of the coke varies with anthracite, between seventy-two and ninety-six per cent.; with caking coals and non-caking coals, between fifty-one and eighty per cent.; with close-burning coals, between sixty and seventy per cent.; and with lignite, between thirty and fifty per cent.

c. Determination of the ash.—The combustion of the weighed quantity of coal, one hundred to one hundred and fifty milligr., or, still better, the finely-pulverized coke from the former, is made in a clay or platinum capsule, which is put into the coal-holder as for the roasting of copper ores (p. 408). If the coal is used, a little is apt to be carried off as dust during the beginning of the operation, so that it is advisable to cover the capsule at first, and not remove the cover until the evolution of gas ceases. By using a weighed platinum capsule, the ash can be directly weighed in the same, without first transferring to the scale-pan, in which operation a loss might easily ensue. The residue in the capsule should be frequently stirred up with a piece of bent platinum wire, in order to bring to the surface all the partially burnt particles of coal.

according to the amount of sesquioxide of iron present, which is formed during the combustion by the oxidation of the pyrites in the coal. An alkaline reaction of the moistened ash upon litmus-paper shows the presence of lime or alkalies; silicates especially being in the ash when no such reaction takes place. The presence of sulphuric acid, resulting from gypsum, may be easily determined by fusing the ash with soda, according to p. 342, and manganese also, according to p. 194.

d. Determination of the absolute heating power by Berthier's process.—Twenty milligr. of the finely-pulverized fuel are mixed in a clay crucible with about five hundred milligr. of finely-pulverized oxychloride of lead, and then covered with the same quantity of the latter. The oxychloride of lead is preferable to litharge on account of its easier fusibility, and is prepared by fusing in a porcelain crucible, over a spirit-lamp or gas-burner, three parts by weight of litharge and one part chloride of lead. After a mixture of eight spoonfuls of borax-glass and one of powdered glass has been added as a cover, the crucible is closed with a clay capsule and put into the coal-holder, which must also be provided with a coal cover. The fusion is then conducted exactly as for a lead or bismuth assay, and is finished in six to eight minutes. If the temperature has been sufficient, the reduced lead button may be seen through the fluid and perfectly transparent slag lying upon the bottom of the crucible. The weight of the button when cleaned from the slag, divided by twenty, gives the quantity of lead that one part of the fuel under examination can reduce; and since one part of carbon reduces thirty-four parts of lead, the heating power of the fuel may be easily ascertained.

The amount of lead reduced by one part of coal varies, with the different pit coals, between twenty-one and thirty-two parts; with the lignites, between sixteen and twenty-five parts.

This assay, the same as that proposed by Domeyko, is very simple and exact.

A glass tube about three lines in diameter, and seven to eight inches long, of not too thin glass, is bent as shown in Fig. 78, and closed at one end, leaving the shorter arm, *a*, one and a half to two inches long. The tube is thoroughly dried, and then from five hundred to three thousand milligr. of finely-powdered ore, according to its richness, intimately mixed with five to ten grm. litharge, being introduced into it, the lower end is gradually heated over the spirit-lamp until the whole mass is fused and the glass begins to soften. The moisture that may be present condenses in the middle of the tube, while the mercury will settle as a thin film, sometimes scarcely perceptible, upon the sides of the glass. When all of the mercury has been sublimed, the tube is carefully heated so as to concentrate the mercury as much as possible to a ring at *b*; the tube is allowed to cool, cut off with a file close to the ring, and the mercury then brushed together to one drop and transferred to a weighed capsule.

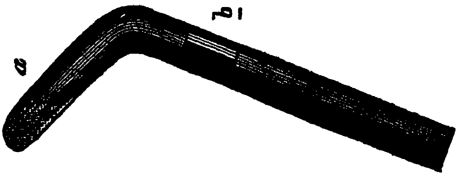

Fig 78.

In this way 0·05 per cent. of mercury can be very readily determined, and the nature of the gangue has no influence upon the result. The excess of litharge serves not only to oxidize the sulphur and selenium, but also to remove the arsenic, antimony, and bitumen so frequently found in ores of mercury, and the resulting metal is so pure that it can be very easily and perfectly united into one drop.

TABLE OF ATOMIC WEIGHTS.

NAMES OF THE ELEMENTS.	SYMBOLS.	ATOMIC WEIGHTS.		NAMES OF THE ELEMENTS.	SYMBOLS.	ATOMIC WEIGHTS.	
		O=100	H=1			O=100	H=1
Aluminium	Al	171·0	13·6	Molybdenum	Mo	575·0	46·0
Antimony	Sb	1504·0	120·3	Nickel	Ni	362·5	29·0
Arsenic	As	937·6	75·0	Niobium	Nb	510·0	48·8
Barium	Ba	857·2	68·6	Nitrogen	N	175·0	14·0
Bismuth	Bi	2600·0	208·0	Osmium	Os	1250·0	100·0
Boron	B	137·5	11·0	Oxygen	O	100·0	8·0
Bromine	Br	1000·0	80·0	Palladium	Pd	662·5	53·0
Cadmium	Cd	700·0	56·0	Phosphorus	P	387·5	31·0
Cæsium	Cs	1641·9	123·3	Platinum	Pt	1232·0	98·9
Calcium	Ca	250·0	20·0	Potassium	K	487·5	39·0
Carbon	C	75·0	6·0	Rhodium	Rh	651·9	52·2
Cerium	Ce	575·0	46·0	Rubidium	Rb	1067·5	85·4
Chlorine	Cl	443·2	35·4	Ruthenium	Ru	651·9	52·2
Chromium	Cr	329·0	26·3	Selenium	Se	495·4	39·5
Cobalt	Co	375·0	30·0	Silicium	Si	175·0	14·0
Copper	Cu	396·6	31·7	Silver	Ag	1350·0	108·0
Didymium	Di	604·5	48·3	Sodium	Na	287·5	23·0
Erbium	E	——	56·3	Strontium	Sr	546·8	43·7
Fluorine	F	237·5	19·0	Sulphur	S	200·0	16·0
Glucinum	Be	87·5	7·0	Tantalum	Ta	860·0	68·8
Gold	Au	2458·7	196·7	Tellurium	Te	806·2	64·5
Hydrogen	H	12·5	1·0	Thallium	Tl	2550·0	204·0
Indium	In	463·4	37·0	Thorium	Th	743·8	59·5
Iodine	I	1587·5	127·0	Tin	Sn	735·3	58·8
Iridium	Ir	1237·5	99·0	Titanium	Ti	301·5	24·1
Iron	Fe	350·0	28·0	Tungsten	W	1150·0	92·0
Lanthanum	La	580·0	46·4	Uranium	U	750·0	60·0
Lead	Pb	1294·5	103·5	Vanadium	V	856·8	68·5
Lithium	Li	87·5	7·0	Yttrium	Y	437·5	35·0
Magnesium	Mg	150·0	12·0	Zinc	Zn	406·6	32·5
Manganese	Mn	337·5	27·0	Zirconium	Zr	558·5	44·7
Mercury	Hg	1250·0	100·0				

A LIST OF MINERALS WHOSE CHARACTERISTICS ARE BUT PARTIALLY KNOWN, ALPHABETICALLY ARRANGED.

Adamite, according to Dana, decrepitates feebly in the closed tube, yields a little water, and becomes white and porcelaneous. On coal a zinc coat and feeble arsenic odour, and in the closed tube with soda and charcoal a ring of arsenic. With borax in O. F. a pearl-yellow bead, colourless on cooling.

Carrollite behaves like niccoliferous linnæite, but the roasted mineral also reacts for copper with the fluxes. (Dana.)

Emplectite and *wittichenite* behave like bismuthinite. The residue on coal yields a copper button with soda or neutral oxalate of potassa.

Eusynchite, *aræoxene*, and *vanadite* (*Vanadit*) are said to give the same blowpipe reactions as the other vanadates of lead.

Kerstenite decrepitates slightly in the closed tube, fuses at redness to a black fluid mass and gives off a very little selenium; at a higher heat some selenous acid. On coal fuses very readily to a black slag, evolves a strong selenium odour and affords lead buttons. The assay is surrounded by a lead coat and beyond this a selenium coat. With the fluxes gives reactions for iron and copper.

Lamprophanite yields water. With soda on charcoal yields metallic lead and a hepatic mass. (Dana.)

Petzite.—Its blowpipe characteristics are not well known; probably it behaves very much like Hessite.

b. Pyrochlore, according to Berzelius, behaves as follows (*vide* p. 146):

Alone it becomes light brownish-yellow, remains lustrous, and fuses with great difficulty to a blackish-brown slaggy mass. Dissolved by borax in the O. F. to a reddish-yellow clear glass, which can easily be made opaque by flaming, and is then yellow; with more the bead becomes opaque of itself on cooling. In the R. F. it becomes dark-red and can be flamed to a light grayish-blue enamel, sometimes with streaks of pure blue.

In S. Ph. it dissolves perfectly, at first, with some effervescence to a bead, which in the O. F. is yellow while hot, but on cooling fine grass-green (uranium). In the R. F. this green becomes gradually dirtier and after a short reduction a dark-red bead, inclining to violet, is easily obtained, as from ferriferous titanic acid.

With soda and nitre on platinum foil gives a manganese reaction.

c. Pyrochlore from Brevig reacts quite like the preceding.

Sartorite, nearly like dufrenoysite, but decrepitates strongly. (Dana.)

Schwartzembergite, according to Dana, is very fusible and loses its colour in fusing. On coal yields metallic globules; in a matrass abundant violet vapours of iodine.

Selenquecksilber kupferblei (Hg, Cu, Pb, and Se) yields in the matrass selenide of mercury. On coal affords a lead coat and a residue which reacts strongly for copper.

Szaibelyite yields water. B. B. splits open, glows, and fuses to a pale, horn-like, brownish-gray mass, colouring the flame yellowish-red. (Dana.)

Wehrlite behaves like tetradymite containing sulphur, and Joseite does the same, but affords the selenium reactions distinctly.

APPENDIX.

I. The employment of quantitative assays with the blowpipe for the determination of various substances in quantitative chemical analysis.

THE method of Websky consists in decomposing the combinations of the elements obtained by analytical methods, with metallic salts, in the wet way, so that the element will be combined with the metal in known stoichiometrical proportions. The amount of metal in the preparation thus obtained is then determined by the blowpipe, and the percentage of the first-mentioned element in the original substance found by calculation, or in other words, the determination of the element is accomplished by a quantitative assay.

Although this method may appear to be very complicated in principle, it may still, under certain circumstances, be of advantage to those who are skilled in assaying with the blowpipe, especially when only approximate results are required, and but small quantities of the substance are at hand.

Among the metals used for substitution, only silver and its combinations have proved successful, except in the determination of quicksilver, where gold is better. The author has properly refrained from describing the use of other metals to be determined quantitatively with the blowpipe, because the method is either too complicated, or wanting in accuracy.

In most cases, the nitrate of silver is used, and less frequently the sulphate or acetate. In altering the various solutions and precipitates to the corresponding silver compounds, and particularly in the treatment of metallic sulphides, it is necessary to observe several precautions, which the author communicates in detail, and the main points of which will be mentioned here.

If a metallic sulphide, soluble in sulphide of ammonium, is under treatment, which dissolves in ammonia with the separation of sulphur, a solution of it in the latter must be filtered directly into a concentrated solution of sulphate of silver in excess. The latter is then heated to boiling, and the black precipitate of sulphide of silver allowed to settle, which requires an hour or two. After filtering and washing, it is assayed for silver. The entire amount of sulphate of silver must be used at once, because the solutions of the metallic sulphides in ammonia oxidize rapidly, and then give less sulphide of silver. For this purpose the amount of dry sulphate of silver necessary, being approximately, but amply estimated, is weighed out and dissolved. 100 parts $\mathrm{Ag\ S}$ contain 69.2 Ag.

The metallic sulphides that are insoluble in sulphide of ammonium are dissolved in sulphuric or nitric acid, hydrochloric acid not being so good, and precipitated from the solution by ammonia and sulphate of ammonium in excess. The solution is filtered, and the precipitate and filter are then treated with nitrate of silver, again filtered, and the residue cupelled. In hydrochloric acid solutions, the precipitates are apt to contain some chloride of ammonium, which increases the resulting silver button. The sulphide of ammonium must, if possible, be freshly prepared, but not too concentrated, except when the sulphides of antimony and arsenic are to be dissolved out; the alteration of the freshly-precipitated sulphides into sulphide of silver must not be done with the application of heat, because in this case the sulphide of silver is decomposed by the nitrates of the metals.

If sulphides are being treated which oxidize very easily, as the sulphides of iron, manganese, or zinc, the precipitate upon the filter must always be covered with water, until it is entirely washed. When the precipitate is very small, it is better to pour it, together with the last addition of wash water, directly into the glass in which it is to be treated with the salt of silver, the filter being pressed down into the fluid, to which the solution of the salt of silver is afterward added.

In many cases, it is necessary to treat both filter and precipitate with the salt of silver, as the latter cannot always be separated from the paper by a jet of water. For this purpose the filter, cut as small as possible, is put into a platinum crucible about two inches deep, and pressed with a glass rod into a paste, a little of the silver solution having been added. The necessary amount of the salt of silver is then added, heat applied, if admissible, and the solution diluted, allowed to settle and then filtered. The particles of paper in the residue remaining in the crucible are then pushed above the level of the fluid mass with a glass rod, and the entire precipitate poured upon the filter. The paper in the crucible is then worked with the glass rod, after adding a little distilled water, and again pushed up on the sides of the crucible, and the fluid poured upon the filter. This manipulation is repeated until all the paper has been finely divided and brought upon the filter.

When the last portions of the wash water have run off, the filter is laid upon blotting-paper, and the outside fold opened, so that the filter takes the shape of a semicircle. A piece of blotting-paper is then laid upon it to soak up the moisture, and it is finally pressed with the hand between dry filter-paper. It is now put into a porcelain dish upon a piece of writing-paper and heated until perfectly dry, after which it is held with the forceps, and burnt over the agate mortar. The residue is triturated with the necessary amount of borax-glass, and put into a paper cylinder with test lead, as described under the silver assay.

The author recommends that the paper for the cylinders be prepared in the following way. It is first soaked in a weak solution of carbonate of soda, and when dry, in a concentrated solution of borax, because the ordinary soda-paper burns off too rapidly for the charge, which frequently, as in the present case, contains considerable carbon that burns with difficulty.

The fusion and the cupellation are performed exactly as in the assay for silver, and from the weight of the resulting silver button the amount of the substance sought is calculated. In this assay, of course, the cupellation loss must be always added.

In this way the author has described methods for determining phosphorus, chlorine, the fixed alkalies and alkaline earths, chromium, uranium, manganese, arsenic, antimony, tellurium, bismuth, zinc, cadmium, lead, iron, copper, and mercury. In the following substances, which are of special interest, the determination can, according to the communicated experiments, be accomplished with accuracy.

Phosphorus.—This is precipitated in the usual manner as basic phosphate of ammonia and magnesia, and the precipitate and filter treated, without warming, with a solution of nitrate of silver. 100 Ag correspond to 9.69 phosphorus, or 22.04 $\overset{\cdot\cdot\cdot\cdot\cdot}{P}$. This method includes the determination of magnesia, which is precipitated from its solution and treated in the above way. 100 Ag correspond to 12.36 Mg O.

Chlorine.—This is combined with sodium or potassium by fusion with the carbonates of the latter, the solution saturated to excess with nitric acid, and chloride of silver precipitated by adding nitrate of silver. The precipitate contains one equivalent of chlorine and one of silver, or 100 Ag correspond to 32.84 Cl.

Potassa, soda, lithia, baryta, strontia, and lime are converted into chlorides by treatment with hydrochloric acid, then heated to low redness, dissolved, and the chlorine precipitated by nitrate of silver. The determination of the chlorine gives at the same time that of the alkalies and earths. For one equivalent of the alkali or the above-mentioned earths, one equivalent of silver is obtained, or 100 Ag correspond to 36.25 K = 43.66 K, 21,46 Na = 28.87 $\dot{\mathrm{N}}$a, 6.08 Li = 13.48 $\dot{\mathrm{L}}$i, 40.45 Sr = 47.86 $\dot{\mathrm{S}}$r, 63.48 Ba = 70.89 $\dot{\mathrm{B}}$a, 18.64 Ca = 26.05 $\dot{\mathrm{C}}$a.

If from a mixture of chloride of strontium and chloride of barium, or chloride of potassium and chloride of sodium, the weight of the freshly-ignited chlorides has been found, and then the chlorine determined by chloride of silver, the quantitative relation of the two substances to each other can be ascertained by calculation.

Copper.—This is precipitated by sulphide of ammonium from an ammoniacal solution of a copper salt, and the precipitate and filter treated as described above, at a very moderate heat, with nitrate of silver, upon which the alteration into sulphide of silver results. For one equivalent of copper one equivalent of silver is obtained, or 100 Ag correspond to 29.3 copper = 36.6 $\dot{\mathrm{C}}\mathrm{u}$.

Iron.—When sesquioxide of iron is precipitated from its solution by means of ammonia and sulphide of ammonium, sesquisulphide of iron, $Fe^2 S^3$, results, which, treated according to the method described, precipitates as Ag S from a solution of nitrate of silver one and a half equivalent of silver for one equivalent of iron. 100 Ag correspond to 17.31 Fe, or to 22.25 Fe, or to 24.72 $\bar{\mathrm{F}}\mathrm{e}$.

Manganese.—This is precipitated from any salt of its protoxide by ammonia and phosphate of soda or of ammonia, and the precipitate and filter treated, without warming, with a neutral solution of nitrate of silver. Exactly one and a half equivalent of silver to one equivalent of manganese is the ratio obtained. 100 Ag correspond to 17.03 Mn, or to 21.97 Mn, or to 24.43 $\bar{\mathrm{M}}\mathrm{n}$.

Arsenic.—This can be determined in two ways. According to one method, pentasulphide of arsenic is formed, the solution filtered, and the precipitate and filter treated in dilute ammonia and gently warmed, the excess of sulphur remaining undissolved. The solution is then filtered directly into a solution of sulphate of silver in excess. The filtration finished, the dark-coloured silver solution is quickly heated to boiling, and the precipitate allowed to settle, which requires an hour or two. It contains five equivalents of silver to one equivalent of arsenic, or 100 Ag correspond to 13.89 As = 18.34 $\dot{\mathrm{A}}\mathrm{s}$=21.3 As.

According to the other method, arsenate of potassa is formed by fusing the substance with nitre and carbonate of potassa. The mass then dissolved, saturated with nitric acid, and highly diluted, if sulphuric acid is present. Nitrate of silver in excess is added, and then ammonia, until the precipitate is redissolved, after which the fluid is evaporated, without boiling, to drive off the excess of ammonia; when there is no longer any smell of the latter noticeable the precipitated arsenic of silver is filtered off. Three equivalents of silver are thrown down for one equivalent of arsenic, or 100 Ag correspond to 23.15 As, or 35.5 As.

Chromium.—Chromate of potassa is formed by fusion with nitre and carbonate of potassa, the mass dissolved in water, saturated with acetic acid, and the solution boiled to drive off the carbonic acid. Water is then added to prevent the separation of acetate of silver, and afterwards nitrate of silver in sufficient quantity. The precipitated chromate of silver contains one-half equivalent of silver to one equivalent of chromium, or 100 Ag correspond to 48.69 Cr = 70.92 Ꞓr, or 93.15 Cr. As the slagging of a large quantity of chromium in the silver assay requires so much borax-glass that the mass cannot be conveniently managed with the blowpipe, the precipitate and filter must be boiled with very dilute hydrochloric acid, by which operation the chromate of silver is altered to chloride, the chromic acid being dissolved. The chloride of silver is then filtered off and assayed.

II. Qualitative Tests.

Test for potassa.—According to Mr. Cornwall, permanganate of potassa in solution furnishes a medium far more transparent to the rays from the potassa flame than indigo solution. It is used in exactly the same way as the indigo solution, and may be advantageously replaced by aniline colours dissolved in alcohol, which offer the advantage of remaining clear. The aniline solution must be of precisely the same colour by *transmitted* light as the permanganate solution. If too blue, some drops of a solution of red aniline are added, and *vice versâ.* Silicates decomposed by acids are tested after such decomposition. If not decomposed, they may be heated on platinum wire with two parts gypsum and one part fluor-spar.

Test for bismuth.—Von Kobell's test for bismuth is the most characteristic. Heat on a large coal the bismuth compound with a mixture of equal parts of sulphur and iodide of potassium. The presence of bismuth is shown by a volatile, bright-red coat, at a distance from the assay: there may be a yellow coat of oxide of bismuth nearer the assay. A large amount of lead obscures the reaction, by causing a copious yellow coat. Mr. Cornwall says that a very small amount of bismuth with a great excess of lead can still be detected as follows. The assay is heated in an open tube four inches long and at least one-third of an inch wide, over the spirit-lamp, with an equal volume of a mixture of one part iodide of potassium and five parts sulphur. Lead alone will then cause a copious sublimate, pure yellow when cold, while the presence of bismuth will cause a red, or if very little bismuth is present, an orange-red band, about one-third of an inch above the lower edge of the yellow lead sublimate. Should an excess of antimony obscure this reaction, remove most of the antimony, in case of sulphides, by alternate treatment in O. F. and R. F. on coal; in case of oxides, fuse them with an equal volume of sulphur in a deep cavity, and then treat the remaining sulphides as before. It will, however, seldom be necessary to test in the glass tube. Care should be taken not to mistake for the bismuth sublimate a sublimate of iodine which may form in the upper part of the tube, but at a greater distance from the assay.

NOTES BY THE EDITOR.

Too much reliance should not be placed on the use of the ivory scale, or of other instruments for measuring silver buttons, unless the operator is pressed for time or only an approximate assay is needed. It is always best to weigh out duplicate assays when the ore is suspected to be poor in silver; if the resulting button is large enough to weigh, the other assay need not be proceeded with. Both Lingke and Oertling make balances which indicate $\frac{1}{1000}$th of a grain with precision, which is near enough to weigh almost any silver button from one assay-centner of ore: if not, a duplicate must be taken, and the two melted together and then weighed. Kleritj invented another instrument for measuring buttons, which consists of an ingenious arrangement of a movable wedge and Vernier scale. The instrument however is not in general use; but as some persons may possess it, I mention it here.

To use the instrument for determining the weight of silver buttons Kleritj has prepared tables, which are here copied in part, and in which the weight of the button, or the percentage of silver, is given for any button from 0.01 to 1 millim. in diameter, obtained from one blowpipe assay centner. The formulæ by which these tables were calculated were given in Nos. 1 and 2 of the *Berg. und Hüttenm. Zeit.* Theoretically the weight of a body is expressed by the equation $G=V\gamma$, in which G denotes the absolute weight, V the volume, and γ its specific gravity. For a sphere $V=\frac{\pi}{6}d^3$, and as the specific gravity of silver is 10.474, we have $G=5.482\ d^3$, or if the diameter of the button was exactly 1 millim., its weight would be 5.482 milligr. But Kleritj says this does not represent the true weight, and that for $\frac{\pi}{6}\gamma$ we must substitute $\mu=6.123d^3=\mu d^3$. The same table may be used for determining the weight or percentage of silver buttons by Rueger's instrument, after determining the diameter in millim.

TABLE FOR DETERMINING PERCENTAGE OF SILVER BY KLERITJ'S OR RUEGER'S INSTRUMENT.

DIAMETER IN MILLIMS.	WEIGHT OR PERCENTAGE $= \mu d^3$	DIAMETER IN MILLIMS.	WEIGHT OR PERCENTAGE $= \mu d^3$	DIAMETER IN MILLIMS.	WEIGHT OR PERCENTAGE $= \mu d^3$	DIAMETER IN MILLIMS.	WEIGHT OR PERCENTAGE $= \mu d^3$
0·01	0·000006	0·26	0·107618	0·51	0·812222	0·76	2·687850
0·02	0·000049	0·27	0·120519	0·52	0·860943	0·77	2·795352
0·03	0·000165	0·28	0·134412	0·53	0·911574	0·78	2·905682
0·04	0·000392	0·29	0·149334	0·54	0·964152	0·79	3·018878
0·05	0·000765	0·30	0·165321	0·55	1·018714	0·80	3·134976
0·06	0·001323	0·31	0·182410	0·56	1·075297	0·81	3·254013
0·07	0·002100	0·32	0·200638	0·57	1·133937	0·82	3·376026
0·08	0·003135	0·33	0·219850	0·58	1·194671	0·83	3·501052
0·09	0·004464	0·34	0·240658	0·59	1·257536	0·84	3·629126
0·10	0·006123	0·35	0·262524	0·60	1·322568	0·85	3·760287
0·11	0·008150	0·36	0·285675	0·61	1·389805	0·86	3·894571
0·12	0·010581	0·37	0·310148	0·62	1·459282	0·87	4·032014
0·13	0·013451	0·38	0·335981	0·63	1·531038	0·88	4·172653
0·14	0·016802	0·39	0·363210	0·64	1·605108	0·89	4·316525
0·15	0·020665	0·40	0·391872	0·65	1·681529	0·90	4·463667
0·16	0·025080	0·41	0·422003	0·66	1·760333	0·91	4·614115
0·17	0·030082	0·42	0·453641	0·67	1·841572	0 92	4·767907
0·18	0·035709	0·43	0·486821	0·68	1·935267	0·93	4·925078
0·19	0·041998	0·44	0·521581	0·69	2·011460	0·94	5·085666
0·20	0·048984	0·45	0·557958	0·70	2·100189	0 95	5·249707
0·21	0·056705	0·46	0·595988	0·71	2·191489	0·96	5·417239
0·22	0·065198	0·47	0·635708	0·72	2·285398	0·97	5·588297
0·23	0·074498	0·48	0·677155	0·73	2·381951	0·98	5·762917
0·24	0·084644	0·49	0·720365	0·74	2·481186	0·99	5·941141
0·25	0·095672	0·50	0·765375	0·75	2·583141	1·00	6·123000

Note 2, to page 162.—"*The silicates under a and β, and the alumina-bearing silicates enumerated under the preceding alkalies and earths.*"

These are the various anhydrous and hydrous silicates, such as Cyanite, Andalusite, Kaolin, Chrome Ochre, Smectite, etc.; as well as the various alumina-bearing silicates of Potassa, Soda, Magnesia, Baryta, etc.

Note 3, to page 3.—The mechanical blowpipe, Fig. 3, is very useful when a *strong* blast is required, as in fusing together substances in clay crucibles, in the square coals, in roasting, and other tedious operations. In the silver quantitative assay, it should only be used in the concentration of the silver lead, and the mouth blowpipe in the final cupellation. Whenever a *fine* oxidizing flame is required, it is always better to use the mouth blowpipe.

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